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IMPROVEMENT OF MANUFACTURING TECHNIQUES AND QUALITY OF OPTICAL SCRATCH STANDARDS FOR FIRE CONTROL SYSTEMS

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This project was accomplished as part of the U.S. Army's Manufacturing Methods and Technology Program. The primary objective of this program is to develop, on a timely basis, manufacturing processes, techniques, and equipment for use in production of Army materiel.

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Laser scribe	Electron beam lithography	Chemical etching
Embedded bubble	Radiometric scanning	Ion beam etching
Diamond scribe	Diffraction gratings	Isotropic
Profilometer	Scalar diffraction	MMT-Optical surface quality standard

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Manufacture of the Optical Surface Quality Standards (Scratch and Dig) has been a problem since their introduction. The present technique of diamond scribing the scratch standard is haphazard mainly because there are no controlling dimensions for the scratch. The problem arises because there is no correlation between the physical and the visual parameters of the scratch.

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20. ABSTRACT (cont)

An evaluation was conducted of manufacturing techniques that would reliably reproduce the standards. Additionally, work was coordinated with the National Bureau of Standards who developed a measuring technique and generated a configuration for the new standard.

The manufacturing process developed under this project utilized an electron beam writing instrument to produce the scratch pattern on photomasks. The photomasks were then processed by wet chemical etching to produce the scratch standards. The pattern generated was a 10-line pattern with individual line structure on the order of 1 micron in width. The results demonstrated a correlation of the scratch pattern to its visual appearance. However, the chemical etching produced low yield due to the non-repeatability of the process.

The results, therefore, while demonstrating for the first time a relationship between the physical characteristics and the visual appearance, also indicate that further refinement of the chemical etching portion of the process is necessary before the technique can be considered cost effective.

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INTRODUCTION

The Product Assurance Directorate of the U.S. Army Armament, Munitions and Chemical Command (AMCCOM) has the mission to provide industry with the scratch and dig standards which are used in conjunction with MIL-O-13830A for the inspection of optical surface quality on Army fire control materiel. The dig standard is well understood because the designation of a dig on a surface is related to its diameter. The scratch standard is not as well understood and less controlled.

Each scratch standard consists of a clear glass substrate with a single fine groove on one of its surfaces. The groove is produced by either scribing with a single-point diamond tool or by wet chemical etching in a photolithographic process. Five levels of scratch severity are provided. These are designated S-10, S-20, S-40, S-60, and S-80, and are established by a set of master standards located at AMCCOM in Dover, New Jersey. The sets of submaster standards which are provided to industry are certified by a visual comparison with the master standards. Typically, the submaster and master are held at roughly the nearest distance of distinct vision (about 25 cm) and viewed in transmission with no interviewing optics. The AMCCOM submaster standards are used in a similar manner by industrial inspectors to rate the severity of surface scratches on manufactured fire control optics.

Since the introduction of the MIL-O-13830 system of surface quality standards, the fabrication of the submaster scratch standards has always been a low yield, trial-and-error process, with the final inspection based on subjective visual comparison. There has never been a quantitative specification for the configuration and design of the "scratch" artifacts on each standard, for the manufacturing processes used, or for the light scattering effects produced by the artifacts.

The scratch widths that are tabulated in the notes on the Army drawings for the scratch standards are for guidance only. There is, in fact, no direct correlation between the width of the artifact and the apparent scratch intensity because the depth, shape, and micro surface texture all contribute to the scattering phenomena, and these factors remain unspecified.

This research program and a concurrent program (ref 1) conducted at the National Bureau of Standards (NBS) in Boulder, Colorado, have been directed toward solving the fundamental problems associated with the design, fabrication, and acceptance inspection of submaster scratch standards for MIL-O-13830. NBS developed the theory (ref 2) to model the phenomena of light scattering through scratch-type artifacts, then built photoelectric instrumentation to measure and characterize the scattering. The NBS theory was utilized to specify design parameters for a series of scratch configurations which were subsequently fabricated and measured on the NBS equipment. The Decilog program addressed the problems of fabrication and acceptance inspection.

EVALUATION OF SCRATCH FABRICATION TECHNIQUES

A pilot study was conducted to evaluate state-of-the-art techniques which could be used for the fabrication of optical scratch standards. Processes used in the graphic arts, optics, and semi-conductor industries--as well as several unique approaches--were considered. Seven techniques plus a set of the currently used standards were selected for an experimental psychophysical study in which optical surface quality inspectors were asked to rate scratch artifacts made by each of the fabrication methods. The objective of the experiment was to determine--based on the subjective ratings--which, if any, of the artifacts would be unacceptable as scratch standards because they do no resemble a scratch or the current scratch standards? Acceptable techniques were further evaluated on the basis of controllability, reproducibility, and compatibility with the NBS instrumentation.

Description of Scratch Samples

The scratch fabrication techniques which were included in the psychophysical study are described in the following paragraphs.

Molded Plastic

The sample shown in figure 1a was developed by Kodak as an internal standard. Seven scratch artifacts (rated 10, 20, 40, 60, 80, 120, and 160) as well as dig artifacts are molded into a single plastic unit. The scratch artifacts have a raised triangular cross section with smooth surfaces and widths ranging from 0.6 micron for the S-10 to 47 microns for the S-160.

Laser Scribe

The artifacts produced by laser scribing consist of a series of separated or overlapping spots (called kerfs) which result from vaporization of the glass surface from heating by a pulsed laser. Typical artifacts are depicted in figure 1b. Kerf diameters of several microns can be achieved by focusing a frequency doubled, Q-switched Nd-Yag laser onto the surface of the glass substrate. The glass is transparent to this laser frequency (0.532 micron), however, and it must first be coated with an opaque material to transform the laser energy into heat and couple it into the glass. Satisfactory results were achieved by coating with either a black felt tip marker or with a 2,000 angstrom (A) layer of vapor-deposited chrome. Coatings were stripped after scribing. Samples were fabricated on a modified Quantronix Model 604 Laser Scribe and on an Electro Scientific Industries (ESI) Microlase* Wafer Processor. Typical machining parameters are summarized in table 1.

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* Trademark of ESI.

Embedded Fiber

Single plastic-and-glass fibers were cemented between glass substrates as shown in figure 1c. The samples used in the experiment were made from fibers approximately 27 micron and 10 micron which produced strengths approximately equivalent to S-120 and S-20 scratches, respectively.

Embedded Bubble

An air bubble is embedded in a glass substrate, and the substrate is heated and stretched to produce a thin hollow cylindrical artifact. The configuration of the sample is shown in figure 1d. The diameter of the cylindrical void was about 20 microns, resulting in an intensity equivalent to an S-60.

Photolithography and Chemical Etch

Chemically etched artifacts are produced by a photolithographic process. This process generally consists of the following steps:

1. Coating of substrate with a resist film

2. Exposing the resist to a spatial pattern of radiation with a "mask" or direct laser, ion, or electron beam illumination

3. Developing the resist to remove the exposed (positive resist) or unexposed (negative resist) regions of the film

4. Post baking to drive off residual solutions

5. Etching of the substrate by either "wet" (i.e., acids) or "dry" (i.e., plasma etching, ion beam milling, or reactive ion etching) techniques

6. Removing the remaining resist film

The samples used in the experiment consisted of a single line with strengths equivalent to an S-20 and S-80.

Diamond Scribe

Number 10 and number 80 commercial scratch standards produced by diamond scribing were included in the samples. The design is shown in figure 1e.

Army Standard

A set of U.S. Army Certified Scratch Standard was used as a reference for the ratings.

Visual Evaluation of Scratch Samples

Fifteen inspectors from six different government and commercial organizations were asked to rate each of eight scratch categories as to how they would like using the sample artifacts as a comparison standard. Ratings were on a scale of 100 for a perfect standard to 0 for one which would be useless. The ratings were then ranked; that is, the fabrication technique given the highest rating was ranked 1, the next highest 2, down to 8, the lowest ranking of the samples. The mean rankings for the eight categories are shown in table 2.

Statistical analyses of variance and "t" tests were used to determine which differences in ranking could be considered statistically significant; that is, the differences that had a low probability of arising by chance.

The high ratings given to the Army standard and the molded plastic (Kodak) standard are shown to be significant. It is felt that the high ratings may have been biased partly by the fact that the subjects recognized the Army standard and partly that both samples were of high quality with good "packaging." In spite of this bias, however, the conclusion that can be reached is that there was nothing unacceptable about the appearance of these scratch artifacts. This is especially significant for the molded plastic (Kodak) standard because the scratch artifact, itself, has a very smooth uniform surface and acts, in part, like a prism in refracting light. It was believed that the visual appearance of this artifact would be quite different from that produced by diamond-scribed or chemically etched artifacts. This is apparently not the case. These results are in agreement with experience at Kodak where the standard has been used for many years in the inspection of optics.

These results indicate that molding, pressing, or replication of scratch standards with sample geometrical features would be acceptable at least from the standpoint of visual appearance.

The statistical analysis also reveals that the laser-scribed artifact produced by the Quantronix equipment and the chemically etched artifact were rated significantly higher than the others. The low rating given to the ESI laser scribed sample is attributed to the fact that the sample consisted of a thin square piece of plate glass on which five closely spaced lines were scribed. The sample was, therefore, difficult to use. The Quantronix sample was more representative of the desired configuration.

The low ratings given to the embedded bubble-and-fiber configuration were supported by the inspector's comments in which they indicated that these artifacts did not "look like" scratches (e.g., the fiber looked three dimensional) and, therefore, would be difficult to use as a comparison standard.

Considerations of Repeatibility, Control, and Stability

The three fabrication processes (i.e., replication or molding, laser scribing, and etching) which were judged to be visually acceptable were evaluated further to establish the extent to which the procedures, parameters, and characteristics of the respective manufacturing processes could be specified and controlled.

The technology of cast replication of submicron-sized features on diffraction gratings is well developed and can be applied to the manufacture of scratch standards. In the replication process, a master artifact is generated in a metal or glass substrate. An intermediate master consisting of a thin (0.001 in.) epoxy film on a glass substrate is molded from this master and then covered with a thin evaporated metal coating. Replicas of the original master are made from the intermediate master and consist of a similar construction, i.e., an epoxy film on the glass substrate. Each intermediate master can reproduce from 10 to 50 replicas.

The replication of fine detail requires the use of epoxy materials which have long curing times. The materials currently in use are cured for 1 1/2 days. While the production rates are low, the cost is comparable to other techniques because no labor is expended during the curing process. Replicated gratings give no evidence of deterioration or change from aging or exposure to thermal variations all the way from liquid nitrogen temperatures to 50°C (special resins can raise this limit to 200°C) (ref 3). Even the highest vacuum seems to have no effect on replica diffraction gratings, and the same holds true for electron bombardment.

Replication and molding processes have the disadvantages of requiring a master mold unless all features of the artifact can be fully specified. Experience at Kodak indicates that with their configuration, it is extremely difficult to specify and inspect for those mold characteristics which will produce a scratch with a specific apparent visual strength. Thus, it has been found necessary to use a trial-and-error process involving the fabrication of many trial molds in order to create molds which will produce scratches that match the Army standards. If these molds are damaged or lost, the trial-and-error process must be repeated. In this situation, a replication or molding process would require that the government be the keeper of a master set of scratch molds and that it manufacture and provide a matching set of secondary molds for procurement. If the scratch artifact can be completely specified, however, as with the designs developed during this program, the replication process offers a viable alternative.

Artifact specification and process control would be difficult to achieve with laser-scribed artifacts. The individual kerfs have complex cross sectional configurations, the parameters of which are difficult to measure and difficult to relate in a theoretical formulation to either the laser beam profile or the resultant scattering pattern. It would, therefore, not be possible to develop a complete and generally applicable manufacturing specification for this fabrication process. For a specific piece of equipment, control parameters could be

established after a trial-and-error process in which trial scratch artifacts are visually matched against standards. Once the parameters for a specific piece of scribing equipment are established, it would seem reasonable to be able to achieve control and repeatability on that specific piece of equipment. This would suggest, however, that the government be the owner of the scribing equipment and that all scratch standard fabrication be carried out on this unit.

The photolithographic process was considered to have the highest potential for achieving the desired degree of artifact specification, process control, and stability. The artifacts, if properly produced, have uniform and straight sides, thereby permitting the measurement of both line width and depth. In addition, the shallow channels produced by this process are conveniently represented as one-dimensional phase objects in the theoretical scattering models. Such models can then be utilized to evaluate artifact design concepts and to develop artifact and process specifications.

IMPROVED SCRATCH CONFIGURATION CONCEPT

A new configurational concept for the scratch standard submasters was proposed by NBS. This concept is compatible with fabrication by state-of-the-art lithographic techniques and eliminates the deficiencies which are inherent in single-channel configurations and which have created the difficulties and uncertainties which presently exist in artifact specification, measurement, and use. A multi-groove grating was proposed as the artifact for the scratch submasters. With this grating, scratch intensity is a function of the number of grooves in the pattern and the depth of each groove. These gratings, if configured with nonequal groove spacings, have three significant additional attributes. First, they can be made to duplicate the visual appearance of natural scratches and the Army Master Standards. Second, the characteristics of light scattering will be determined primarily by the geometrical parameters (i.e., line width, spacing, depth, and number of lines) of the grating and only minimally by the micro surface texture within the groove. Third, the scattering pattern can be shaped to produce a broad scattering maxima, away from the direction of the specular light.

The latter attribute is important from two standpoints. First, many discrepancies in inspection arise from the fact that the apparent intensity of the single-channel standards increases monotonically as the angle of observation decreases and the line of sight approaches the specular direction. (With reference to figure 2, this is the situation when light rays from the source of illumination fall within the field of view of the eye.) Since an inspector always adjusts his viewing angle to produce a maximum apparent intensity, a broad scattering maxima located away from the specular direction would force each user to position his eye and the standard at the same location with respect to the source and, thereby, view the same region of the scattering pattern.

The second advantage of having a broad scattering maxima is that the intensity of the relative scattering in the region of the maxima can be made to correlate with the apparent visual strength of the artifact. The difficulties created by single channel artifacts are demonstrated in figures 3 and 4, the data for

which were obtained by NBS using the instrumentation described in reference 1. Figure 3 is a superposition of scattered light patterns of five single channel scratch submasters, each rated at S-40. One observes the wide variation in scattering patterns which are obtained from indentically appearing scratch artifacts. The sharp central peak is the undiffracted light. Scattered intensity is normalized relative to the peak. It is usual to view the samples at angles somewhere between 5 and 15 degrees. The variation of scattered intensity measured at 5 and 10 degrees as a function of visually rated scratch number is shown in figure 4. The open circles connected by the line represent the average intensity scattered by scratches with a given designation. The black circles show the highest and lowest values measured. Plotted on the semilogarithmic scale, the average 5 degree data correlate well with scratch number. The data, however, show a great deal of uncertainty, and the data points overlap in such a way that a scratch with a given designation could not always be distinguished from its nearest neighbor with a single photoelectric measurement. The curves in figure 3 suggest, in addition, that even a measurement based on integrated value may not correlate well with the visual rating.

A typical scattering pattern from one of the proposed grating configurations is shown in figure 5. The broad, off-specular maxima is seen in the neighborhood of 8 degrees. This curve was obtained from a computer simulation of the NBS scattering model (ref 2).

The design of candidate grating artifacts was performed by NBS. The primary design requirements were:

1. That the artifacts be comparible with state-of-the-art lithographic processes.

2. That the intensity maxima peak between 5 and 10 degrees, the normal viewing angle for the existing standard.

3. That the scattering produced by the artifact resemble that produced by natural scratches and the Army Master Standards. Specifically, the artifact should appear achromatic.

4. That the overall width of the grating should be below eye resolution at the shortest distance of distinct vision. If the eye were diffraction limited, this would imply that the overall width be no greater than 27.5 μm, assuming a pupil diameter of 5 mm, a viewing distance of 25 cm, and a mean wavelength of 550 nm. Since the eye is not, in reality, diffraction limited, the width can be higher. An upper limit can be obtained by assuming that the resolution limit of the eye is about 30 seconds of arc. This is equivalent to a width of 36 μm at the 25 cm viewing distance.

5. That a set of grating artifacts cover at least an intensity range equivalent to that of the S-10 through S-80. Measurements of scratch submasters by NBS indicate that this is a range, on average, of about 50:1 (fig. 4).

To design the gratings, NBS developed a scalar diffraction theory (ref 2) which was used to calculate the intensity scattered by gratings with identical rectangular grooves with modulated spatial frequency.

For purposes of design, the Fraunhofer-Kirchoff diffraction theory can be applied to the gratings if the individual grooves are wider than a few wavelengths and shallow enough that single scattering may be assumed (ref 4). Likewise, the scattering angles must be sufficiently small that single scattering applies.

The intensity of radiation scattered by a finite grating consisting of identical grooves is equal to the product of a grating interference function G (λ,N) and the well known diffraction function of a single groove or slit. That is for any angle θ,

$$I(\Theta) \propto 4b^2 \sin^2(\Delta/2) \operatorname{sinc}^2\left((\pi/\lambda)\, b \sin\Theta\right).\ G(\lambda,N) \qquad (1)$$

where

b = the width of the groove

λ = the wavelength of light

$\operatorname{sinc}(u) = \frac{\sin u}{u}$ for any function u

Δ = the phase depth of the groove and is equal to

$$\Delta = \frac{2\pi\,(n-1)\,Z}{\lambda}$$

where

n = the index of refraction of the glass substrate

Z = the depth of the groove

The function $G(\lambda,N)$ is a complex function dependent primarily on the groove-to-groove spacing configuration and the total number of grooves, N, in the pattern.

The scattering function $I(\Theta)$ reaches its first minima at an angle of λ/b. (This causes the sinc function in equation 1 to go to zero.) To have the broad maximum occur between 5 and 10 degrees, the first minimum must fall beyond 15 degrees. Assuming, again, a mean wavelength of 550 nm, this requirement sets the maximum allowable groove width at 2 micron.

The term $\sin^2(\Delta/2)$ establishes the relation between scattering intensity and the depth of the groove. For the case of optical glass with $n = 1.5$, the function is maximized when $Z = \lambda$ or at about 0.55 micron. Because of the sinusoidal variation of scattered intensity with depth, the scattered intensity is also least sensitive to depth error when the grooves are 1 wavelength deep. If the grooves are less than this depth, the scattered intensity is reduced by the

factor $\sin^2(\pi Z/\lambda)$, but the overall pattern shape is not changed. Therefore, the scattered intensity may be calculated for any convenient depth and the depth factor may be used to scale to other depths.

By varying the depth and number of grooves in each grating, it is possible to simulate the scattering pattern of any scratch.

The NBS model was used in a parametric study to generate the theoretical scattering profiles of 69 different line spacing configurations. The number of grooves ranged from 1 to 20, each with different variations in the groove spacing geometry. All profiles were run for a 1-micron line width, a 0.1-micron groove depth, and a source of illumination with an angular subtense of 2 degrees at the eye.

Thirteen design configurations plus a single groove artifact were selected for fabrication and evaluation. Selection was made on the basis of two criteria:

1. That the scattering maximum occur between 7 and 8 degrees.

2. That the maximum be as broad as possible to minimize the possibility of color separation. An arbitrary but quantitative criterion was established for this characteristic. A parameter P was defined as the ratio of scattered intensity at 5 degrees to the scattered intensity at the peak. Patterns were selected whose values of P exceeded 0.6.

Line spacing specifications for each of the 14 scratch patterns are shown in figure 6.

The simulated scattering profiles for the 13 multi-channel configurations of figure 6 show that it is not possible to achieve the 50:1 range of scattering intensity by varying only the number of lines in the pattern. A plot of peak-scattered intensity versus number of grooves is shown in figure 7 for groove depths of 0.1, 0.05, and 0.03 micron. It is seen that the maximum achievable range, even using an excessively wide 20-line pattern, is only about 5:1 for any given groove depth. It was concluded, therefore, that all of the scratch standards should, if possible, be made from identical geometrical patterns and that the intensity variation be achieved through variation in the groove depth.

Visual evaluation and scattering measurements on fabricated samples indicated that the 10-groove geometry, identified as pattern no. 6 in figure 6, met all requirements for scratch levels S-20 through S-80 and for intensities considerably greater than that of S-80. It was not possible to produce an S-10 scratch with any multi-groove pattern.

A tabulation of the depths and widths of the grooves on those samples of pattern no. 6 which visually matched the Master Standards at ARDC is contained in table 3. Dimensions were obtained from profilometer scans made on a Rank Taylor Hobson TALYSTEP profilometer. The S-10 sample is a single-groove configuration.

FABRICATION OF SCRATCH SAMPLES

Lithographic processes using both "wet" chemical and "dry" ion beam etching have been utilized to produce samples of the multi-channel scratch standards.

Process Description

The process consists of generating the desired pattern in a layer of chrome which is vacuum deposited onto a glass substrate. The pattern is etched into the glass with the chrome coating serving as a mask. The coating is then stripped away.

The process control requirements for this technique are stringent in that the patterns which are generated in the substrate coating (prior to etching) must have widths on the order of one micron, spacings which are controlled to the submicron level, and uniformity in both width and spacing over the entire 1 1/4-inch length of the pattern. After considerable investigation, it was determined that these requirements could best be satisfied on a repeatable basis by using direct-write electron beam lithography and substrates of a quality consistent with those used for silicon wafer processing in the solid state semi-conductor industry. Optical projection and laser and diamond scribing techniques used in optical reticle production were investigated but these could not satisfy the micron and submicron requirement. The techniques and equipment used in the fabrication of precision diffraction gratings are able to generate long submicron-sized grooves but are not designed to produce grating patterns with non-equal line spacings. The direct-write electron beam approach was selected because it meets all requirements, and services to fabricate substrates with commercial equipment are readily available from several sources. Commercial direct-write electron beam equipment (e.g., Perkin Elmer MEBES II) is computer-controlled, can produce beam spot sizes as small as 0.1 micron, and can maintain positional accuracy of the beam to within 1/8 micron over a distance of 8 inches.

To achieve micron levels of resolution, the mask blanks must be carefully fabricated to maintain flatness (on the order of 1/20 wave) and uniformity of the glass substrate, the chrome coating, and the resist layer. Satisfactory results were achieved with commercially available "master" quality substrates which are precoated with chrome and resist. The chrome and resist layers are about 1,000 A and 5,500 A thick, respectively. The adhesion of the chrome layer to the glass substrates in these products is good, and there has been no evidence of chrome separation during the etching process.

With wet chemical etching, the resist layer is stripped from the chrome prior to etching to minimize the depth of the etching "window" in the mask and to facilitate the flow of etchant in the micro channels. With ion beam etching, the etch rates of the glass and chrome are similar and the chrome mask layer will etch away as the glass substrate is etched. The resist layer is, therefore, left on if groove depths in excess of 1,000 A are to be generated. The ion beam etching was performed on a 3-inch Veeco Ion Etch System operating at a current density of 0.7 ma/cm^2.

Substrates made from white crown glass (often called white soda lime glass and equivalent to Schott B-270) produced smooth and uniform channels with both ion beam and semi-conductor grade, buffered oxide etchant (e.g., J. T. Baker, 9 parts 40% ammonium fluoride, 1 part 40% hydrofluoric acid). This same etchant produced rough-edged grooves when used on substrates made from low expansion glass (e.g., Hoya LE-30 or Corning 7059).

To minimize costs in this program, one mask containing all 14 patterns was generated by direct-write electron beam lithography. Sixty additional replications were then made from it by less costly contact printing processes.

The quality and uniformity of the replicated patterns was not, however, as good as those made by direct writing; and the yield of marginally acceptable products was only about 40%. The micron level geometrics required in these patterns clearly pushes the contact printing process past its limits, and it should not be considered a viable alternative for scratch standards production. However, since all of the S-20 through S-80 submasters can be made from the same pattern geometry, cost reductions can be achieved by placing as many identical patterns as possible onto a single substrate and then slicing up the substrate after etching (fig. 8). Samples of the 10-line pattern were generated in this way by etching 10 identical patterns into 5 inch x 5 inch white crown substrates. The cost of electron beam machining such a configuration in production (quantities of 300 per year) would be about $600 per substrate.

Process Evaluation

The process that is envisioned for production of the scratch submasters is based on an assumption that for any lot of electron beam-generated chrome masks, one can experimentally determine a calibration curve relating etch time to either the groove width and depth or to a measured scattered intensity. One processed substrate containing many identical patterns, for example, might be drawn from a larger lot of substrates and cut up into individual pieces, each containing a single 10-line pattern. Each piece would be etched for a different time and the resulting etched patterns measured geometrically and/or radiometrically. The etch time required to generate each of the standard scratch intensity levels would then be determined from this calibration curve and used to process the remaining lot of substrates. In other words, repeatability of the etching process, at least on a lot-to-lot basis, is a requirement to achieve high product yields. An additional requirement implied by the above process is that all of the identical patterns on a single substrate etch at the same rate to produce similarly appearing artifacts.

During the various stages of investigation of the lithographic process in this program, a wide variation in the etch rate of the buffered oxide etch on white crown glass was observed. These variations occurred, however, with substrates from different supplies, with patterns of differing line widths, and with etchants from a multiplicity of production runs. In all cases, etching was accomplished by immersion of the substrate into a stagnant etchant bath. After a specified time, the substrate was removed from the bath and rinsed with deionized water. Etch rates from 200 to 1,000 A per second were observed.

It is clear that line width will have an effect on etch rates in a stagnant bath because of the line width's effect on the transport of dissolved material away from the etching zone. Spray etching and bath agitation might minimize these effects.

The fact that substrates from different suppliers were used is a factor contributing to variation in etch rate because--at least in terms of the white crown glass--optical properties and, therefore, chemical composition are not controlled as tightly as the other optical glasses. High priced quartz substrates would probably yield higher repeatability and uniformity. It has not, however, been determined if such a step is necessary.

The etch rates experienced during the program are, in general, too high to produce good uniformity. Samples were generated with etch times ranging from 1 to 6 seconds. With this short time, it was not possible to adequately control the etch time or to ensure uniform substrate immersion, at least in the manual process used. The etchant should be diluted to yield etch times of at least 30 seconds. Uniformity is also enhanced by restricting the working area of the substrate to a centrally located circle whose diameter is equal to about 75% of the substrate width (for example, fig. 8).

Time and funding limitations did not permit a full evaluation of the adequacy of the repeatability and uniformity of wet chemical etching.

An experiment was run to evaluate the long-term stability of the finely etched artifacts. One of the substrates containing the 14 different etched patterns was placed into an oven for 2 weeks at a temperature of 350°F (177°C).

The artifacts were rated visually before and after the high temperature exposure. There was no observable change in the apparent scratch intensity. One can extrapolate the two weeks at high temperature to long term room temperature exposure by comparing the viscosity of glass at the two temperatures. At 37°C, the viscosity of soda-lime silica glass is 10^3 to 10^4 poise (ref 5). At 177°C, the viscosity is 10^{12} to 10^{13} poise, a factor of 10^8.

One would not, therefore, expect any appreciable change in the appearance of the standards over a period of many years due to material flow. Surface contamination would most likely be the factor in throwing a standard out of calibration.

It should be noted, in addition, that no difficulty was encountered in cleaning the multi-channel artifacts wih lens tissue and cleaner or in an ultrasonic bath.

EVALUATION OF MULTI-CHANNEL SCRATCH STANDARDS

The samples of the multi-channel scratch standard configurations were evaluated on the basis of the following six criteria:

1. Artifact resembling a scratch and appearing achromatic.

2. Scratch intensity exhibiting a clearly perceived maximum between 5 and 10 degrees viewing angles.

3. Pattern width below the resolution limit of the eye.

4. Demonstrated range of S-10 through S-80 with a single multi-channel pattern.

5. Demonstrated correlation between scratch number and relative intensity of scattered radiation (NBS measurement)

6. Demonstrated correlation between scratch number and groove dimensions.

Visual Evaluation

Each of 377 multi-channel artifacts (representative of the 13 configurations of fig. 6) were visually inspected. They were rated on the basis of criteria 1, 2, and 3 above and assigned a tentative scratch number based on visual comparison with the AMCCOM Master Standards.

None of the multi-channel configurations produced scattering at the S-10 level. The 10-line pattern identified as number 6 in figure 6 was selected for further evaluation because it clearly satisfied criteria 1, 2, and 3. It yielded scattering intensities equivalent to S-20 through S-80; and it had line spacings which were easily reproduced by the lithographic process.

The 10-line artifacts were rated again by the AMCCOM optical inspector who performs the scratch standard certifications. Artifacts which matched either the high or low AMCCOM Masters or fell in-between were selected for radiometric scanning and profile measurement.

The ARDC inspector gave a high rating to the quality of the chosen artifacts and expressed the opinion that the artifacts could not be distinguished in appearance from the AMCCOM Masters when viewed at the angle of maximum scattering. The scattered radiation from these multi-channel artifacts appears bluish when viewed at angles on either side of the maximum but are fully achromtic in the region of maximum intensity. This makes the task of positioning the artifact very easy.

Three 10-line artifacts were etched by an ion beam process. Their appearance was identical to those produced by wet chemical etching and could not be distinguished from them.

Fabrication of scratch artifacts by ion implantation of boron bi-fluoride (BF_2) atoms into the white crown glass substrate was attempted. While the implantation process is believed to be capable of fine control, the samples produced were light and do not appear "scratch like."

Radiometric Measurements

Rated multi-channel and single-channel samples were submitted to NBS (Boulder, Colorado) for measurement of profiles of scattered intensity. The profiles of four chemically etched artifacts and two ion beam-etched artifacts (all with the 10-channel pattern) are given in figures 9 through 14.

The choppy appearance of the scans arises from the use of a coherent light source in the NBS instrumentation. What is observed are the small secondary maxima and minima which occur in all gratings of a finite width but which are not perceived by the eye. The NBS simulation model assumed that the source of illumination had a 2 degree subtense, similar to normal viewing conditions and, therefore, also does not exhibit these secondary effects (fig. 5). One should use the scans of figures 9 through 14 by averaging adjacent maxima and minima. Realizing that the intensities are plotted on a logarithmic scale, an appropriate intensity envelope in the region from 5 to 15 degrees can be obtained by drawing a smooth curve through points located below each peak but which have an intensity of one-half the maximum intensity shown.

It is important to note that even with the coherent illumination, all of the samples exhibit identical scan profiles and differ only in relative intensity. This is the feature which allows the scattering intensity to be characterized by a single radiometric measurement.

The locally averaged and normalized peak intensities measured at the center of the broad scattering peak which occurs at 8 degrees are plotted in figure 15 along with the data of figure 4. An additional data point for an artifact visually rated between an S-40 and an S-60 is shown at the S-50 position. Observe the high degree of correlation demonstrated by both chemically etched and ion beam-etched samples. The data for a single-line artifact, representing an S-10, are also shown. The intensity for this data point was obtained from the measured scatter at 8 degrees, the full profile of which is shown in figure 16.

Geometric Measurements

Profile scans of the seven rated samples were obtained by the NBS National Engineering Laboratory (Washington, DC) using a Rank Taylor Hobson, Ltd. TALYSTEP Profilometer with a 0.1-micron probe. A scan of the chemically etched S-40 sample is shown in figure 17. Observe that the ratio of the vertical-to-horizontal scales on the scan is 25:1 and that the individual grooves resemble shallow channels. Each mark on the vertical scale represents 0.04 micron and each mark on the horizontal scale represents 1.0 micron.

A scaled profile of the TALYSTEP probe is shown inside the first groove to indicate that the probe tip is significantly narrower than the groove. The scan that is generated, therefore, is a true representation of the groove profile. The aspect ratios of all of the groove cross sections are similar to those shown in figure 17, including the two samples etched by ion beam lithography.

Five scans were made across each sample, and an average mean width, average depth, and variances were computed. The results are given in table 3. The mean width is defined as the width of the groove midway between the groove bottom and the upper surface of the substrate.

The existence of a correlation among scattered intensity, scratch number, and the groove dimensions can be investigated by defining a groove geometry parameter which is a function of groove width and depth. Such a parameter is suggested by equation 1. Since all of the S-20 through S-80 samples represent identical line spacing configurations, equation 1 shows that the scattering intensity is dependent, at least theoretically, only on the factor $b^2 \sin^2 (\Delta/2) \operatorname{sinc}^2 \left(\frac{\pi}{\lambda} b\sin\Theta\right)$, since the function $G(\lambda,N)$ is identical for all of the multi-channel samples. For $n = 1.5$ and $\lambda = 0.55$ µm, this factor which shall be represented by symbol F is equal to

$$F = \sin^2 (2.862Z) \sin^2 (0.795b) \tag{2}$$

where Z and b are the groove depth and width, respectively, in microns.

The geometry factor F has been computed for each of the samples itemized in table 4 and the results plotted against the measured peak scattered intensity (fig. 18) and the rated scratch number (fig. 19). A perfect correlation agreeing with the theory of equation 1 would appear as a straight line with a 45 degree slope in figure 18. Such a line has been drawn in the figure and shows this high degree of correlation. The data points in circles are for chemically etched samples; the points in diamonds are for samples etched with ion beam.

A good explanation cannot, at this writing, be offered for the deviation of the ion beam-etched S-20 sample.

The ion beam samples were included to show the effects, if any, of differences in the micro surface texture of the groove. The ion beam sample should have grooves with considerably smoother sides than those of the chemically etched samples. No attempt was made, however, to verify this and the profile scans do not show this level of detail.

One should keep in mind that there are not a sufficient number of samples to determine, with reasonable statistical confidence, the variances produced by the fabrication process. The single wayward data point may not, therefore, be of great statistical significance.

If, however, the correlation between the groove geometry factor F and the measured intensity is accepted for at least the chemically etched samples, one can define a relationship between acceptable groove width and depth for each of the scratch numbers. Based on the measured data, the relationships for the S-20 through S-80 chemically etched artifacts are:

$$\text{S-20} \qquad \sin^2 (2.862Z) \sin^2 (0.795b) = 0.010 \tag{3}$$

$$\text{S-40} \qquad \sin^2 (2.862Z) \sin^2 (0.795b) = 0.020 \tag{4}$$

$$\text{S-60} \qquad \sin^2 (2.862Z) \sin^2 (0.795b) = 0.055 \tag{5}$$

$$\text{S-80} \qquad \sin^2 (2.862Z) \sin^2 (0.795b) = 0.118 \tag{6}$$

The functions in equations 3 through 6 are concentric ellipses as shown in figure 20. Also shown on the graph is a hypothetical curve representing the growth in groove width and depth as a function of etch time. This etching causes an upward slope because wet chemical etching is an isotropic process in which etching progresses down as well as to the sides. The intersection of the characteristic curve with the b axis represents the width of the chrome mask before etching. The etch times represented at the intersections of the etching characteristic with each of the groove geometry functions are the etch times for production of subsequent standards. In actual production, the data for the etching characteristic would be obtained from measurements of artifact samples selected from a production lot.

Observe that the width of the lines in the chrome mask pattern may take on a range of values. The requirements are that the resulting etching characteristic intersect all of the groove geometry function curves, and that the line not be so wide that the etched grooves obliterate the pattern (i.e., greater than 2.8 micron) or so narrow that they approach the resolution limits of the resist (i.e., less than 1/2 micron).

The existence of a correlation between the groove geometry factor and both scattered intensity and scratch number shows that dimensions can serve as one basis for quantitative acceptance inspection of scratch standard submasters. The other basis would be measurements of scattered radiation with a wide (2 degree) subtense source and instrumentation similar to the NBS design. A simplified measuring device is shown in refernce 1.

CONCLUSIONS AND RECOMMENDATIONS

A new design configuration and fabrication process has been developed for the Army Scratch Standard Submasters. The new standards appear visually similar to the existing standards but are easier to use and should provide more consistent inspection results. Complete quantitative specifications can be developed for both the design and acceptance inspection of the standards with both geometric and radiometric measurements.

Some additional testing is required, however, to more fully evaluate the repeatability and uniformity of the wet etch process and determine if the S-10 level can be fabricated with the same pattern configuration as the S-20 through S-80 standards. The following steps are recommended:

1. Determine the etchant dilution needed to reduce the etching rates in white crown glass by a factor of 20 to 30.

2. Use the diluted etchant in determining if an S-10 standard can be fabricated with the 10-line pattern.

3. Obtain additional electron beam-etched masks and generate sets of standards with the processes described in this report. Evaluate the repeatability of the wet etch process from substrate to substrate and the uniformity among the etched artifacts on each substrate. Process the samples with the diluted etchant.

4. Make geometric and radiometric measurements of all samples and verify the correlations between these measurements and the visual scratch ratings.

REFERENCES

1. Matt Young and Eric G. Johnson, Jr., "Objective Measurement and Characterization of Scratch Standards," ARDC Contractor Report ARPAD-CR-84002, National Bureau of Standards, Boulder, CO, June 1984.

2. Eric G. Johnson, Jr., "Simulating the Scratch Standards for Optical Surfaces: Theory," Applied Optics, vol 22, no. 24, 15 December 1983, pp 4056 through 4068.

3. Diffraction Grating Handbook, Bausch and Lomb, Inc., Rochester, NY, 1970.

4. Matt Young, "Objective Measurement and Characterization of Scratch Standards," Proc SPIE, vol 362, 1983, pp 94 through 100.

5. Viscosity - Temperature Relations in Glass, International Commission on Glass, ICG Prague 6, Czechoslovakia, 1970, p 24.

Table 1. Laser scribe characteristics

Laser: Frequency doubled Nd:YAG
Wavelength: 530 A
Pulse width: 60 ns
Pulse rate: 1 kHz

Beam power (mW)	Table traverse speed (mm/sec)	Approximate scratch rating
10	10	10
10	5	20
30	5	low 40
40	5	high 40

Table 2. Ranking of scratch categories

Category	Mean ranking
Molded plastic (Kodak)	2.53
Army standard	2.76
Laser scribe (Quantronix)	4.26
Chemical etch	5.06
Diamond scribe	5.36
Laser scribe (ESI)	5.40
Embedded bubble	5.46
Embedded fiber	5.80

Table 3. Dimensions of scratch standard samples

Scratch level rating[a]	Average mean width[b] (microns)	Standard deviation for width	Average depth[b] (microns)	Standard deviation for depth
$S\text{-}10_L$[c]	0.96	0.042	0.01	0.002
$S\text{-}20_L$	0.71	0.065	0.06	0.005
$S\text{-}20_L$	0.72	0.044	0.08	0.008
$S\text{-}40_L$	0.85	0.079	0.08	0.009
S-40	0.93	0.063	0.07	0.005
$S\text{-}60_L$	0.76	0.084	0.15	0.011
$S\text{-}80_H$	1.12	0.061	0.16	0.013

[a] Subscripts L or H indicate that samples match either the low or high limits of the Master Standards.

[b] Average depth (width) taken at five locations on each of the ten grooves.

[c] Single groove configuration.

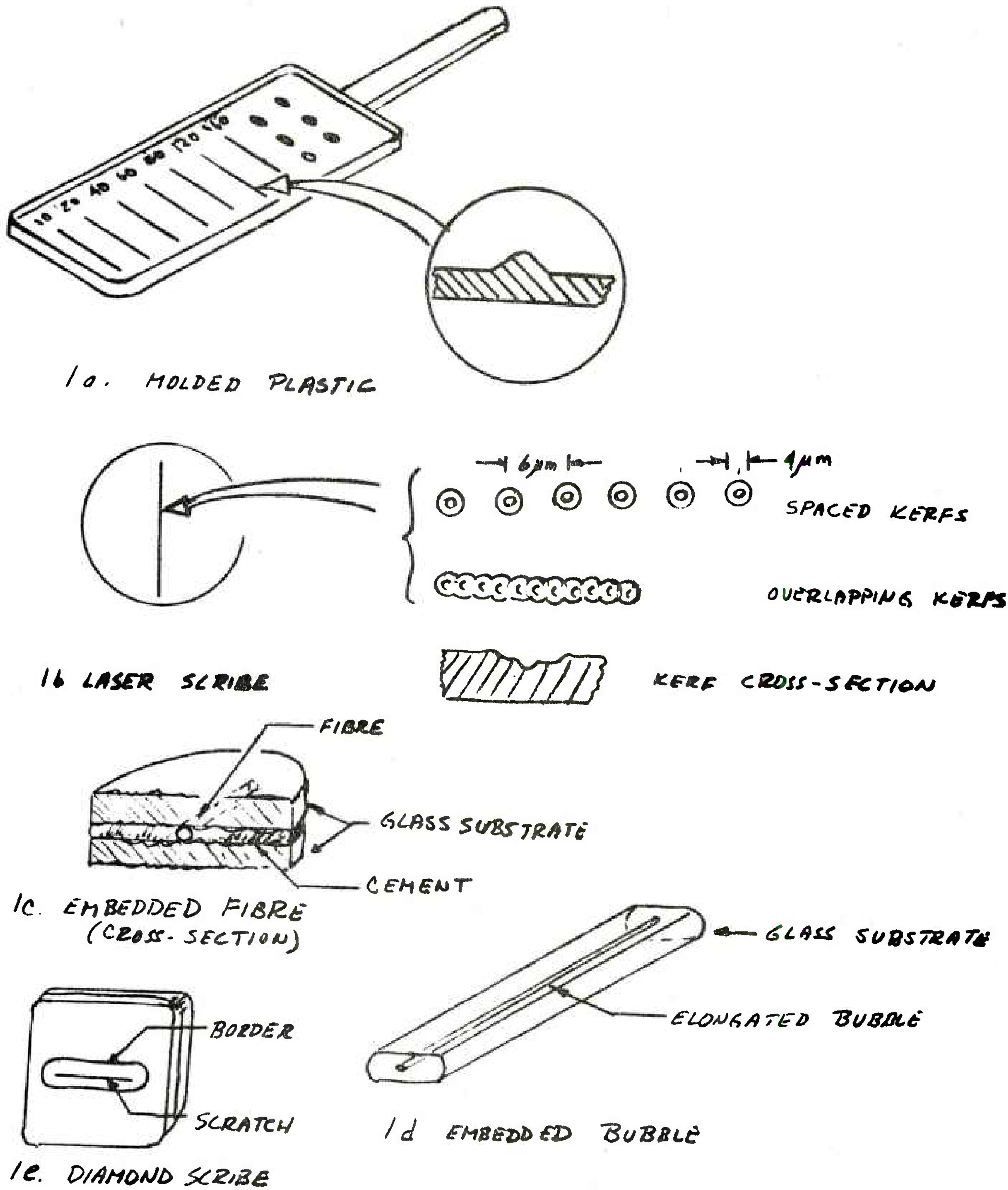


Figure 1. Scratch configurations (not to scale)

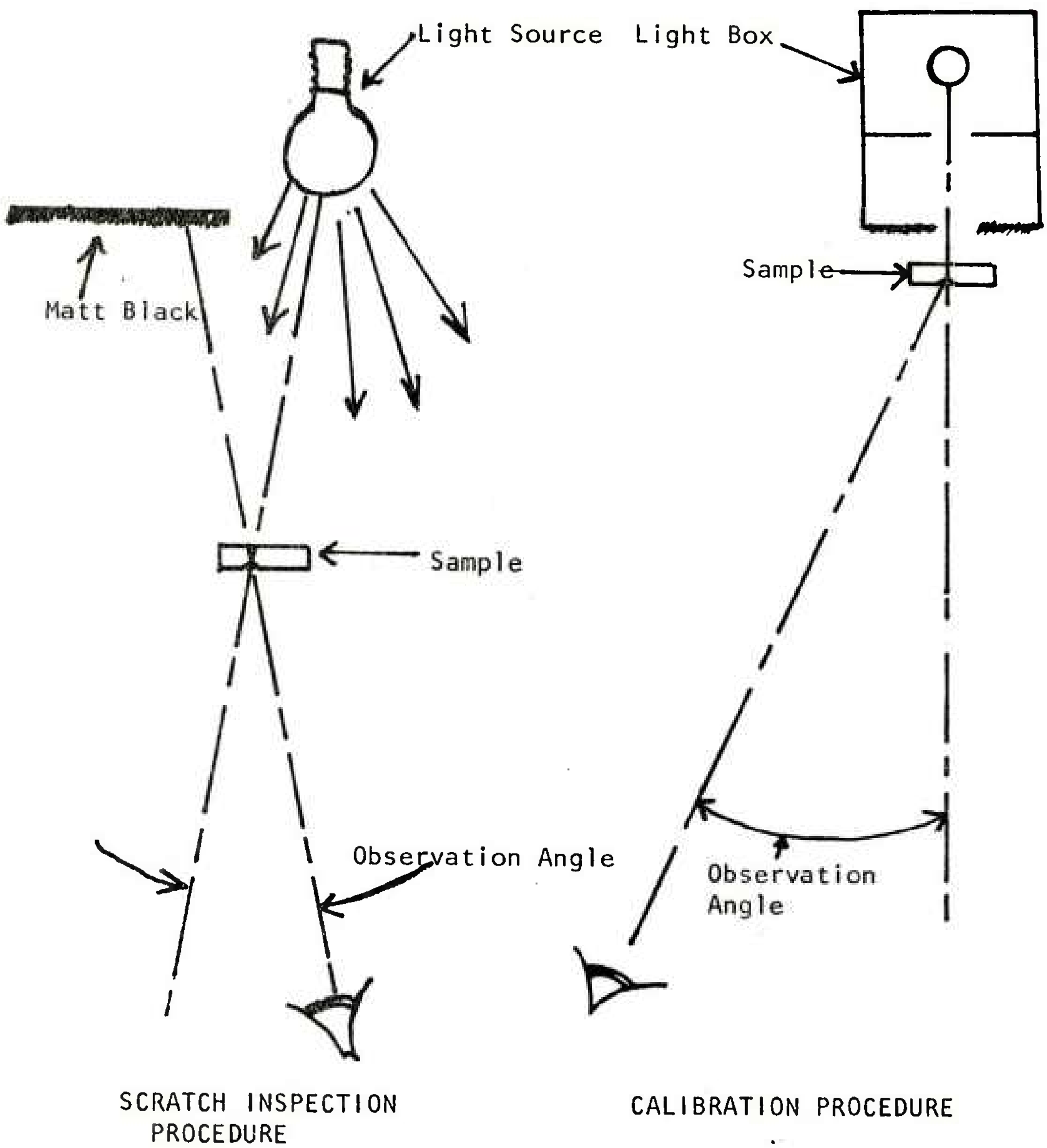


Figure 2. Scratch inspection and calibration procedures

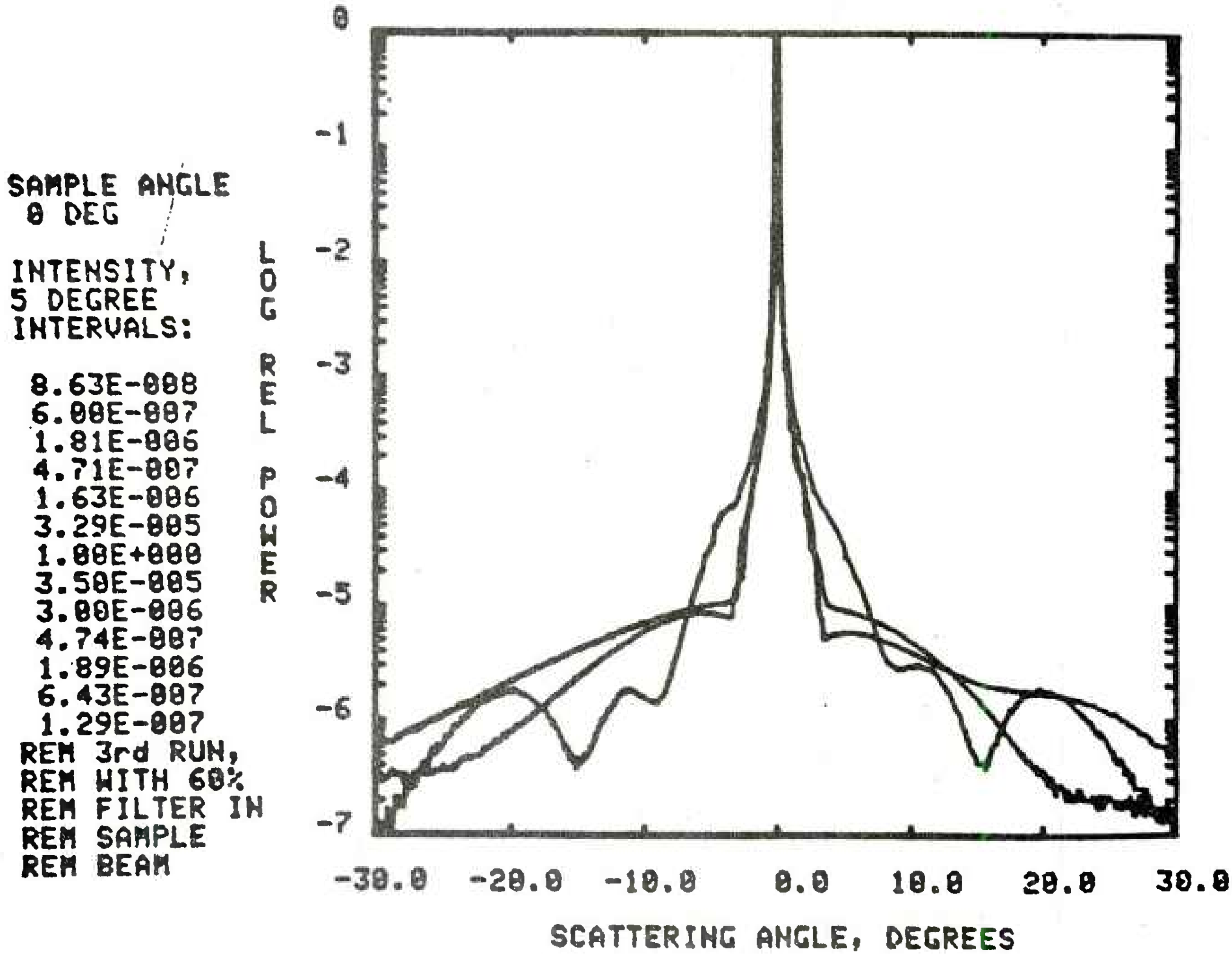


Figure 3. Comparison of relative scattered intensity patterns for S-40 scratch submasters

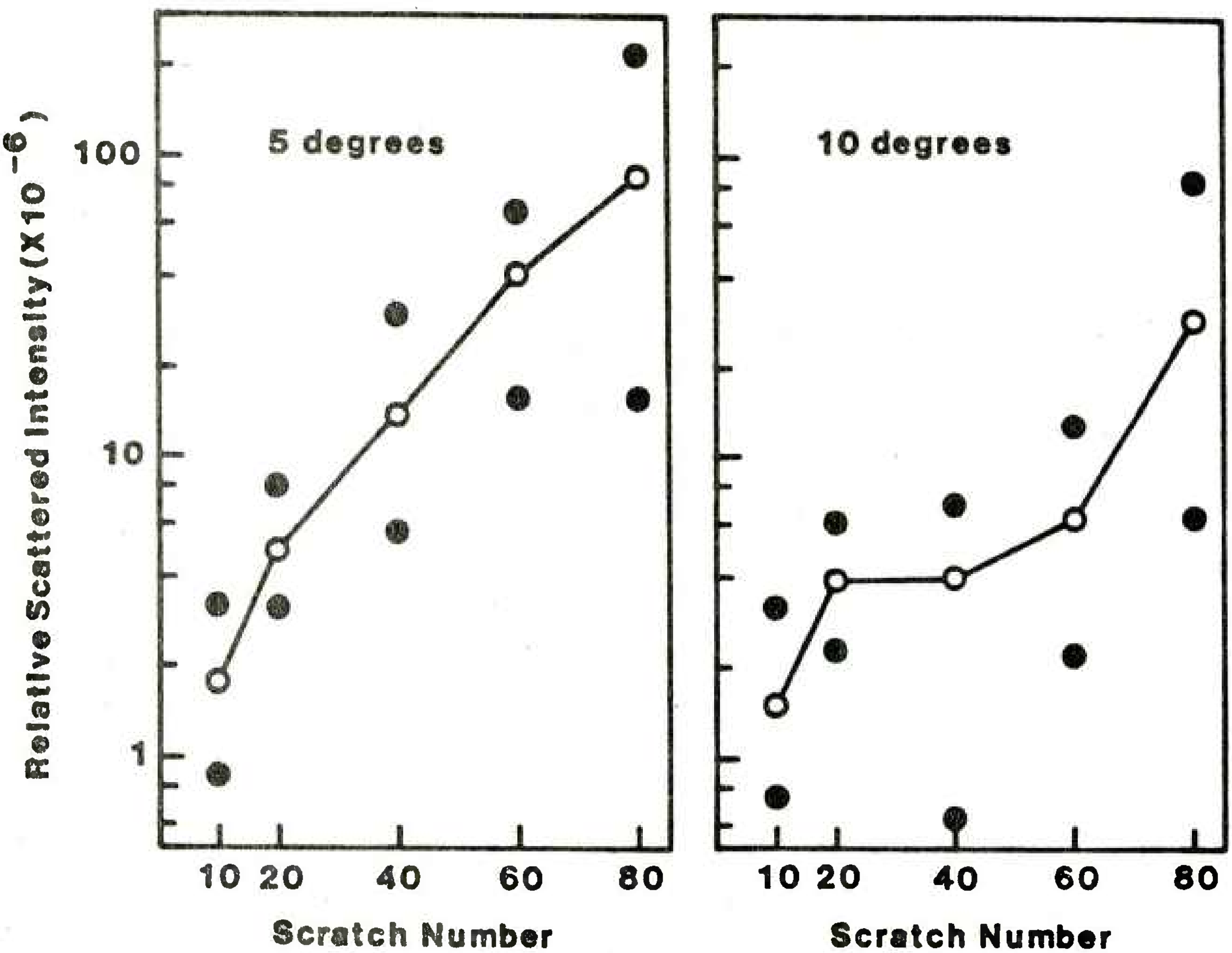
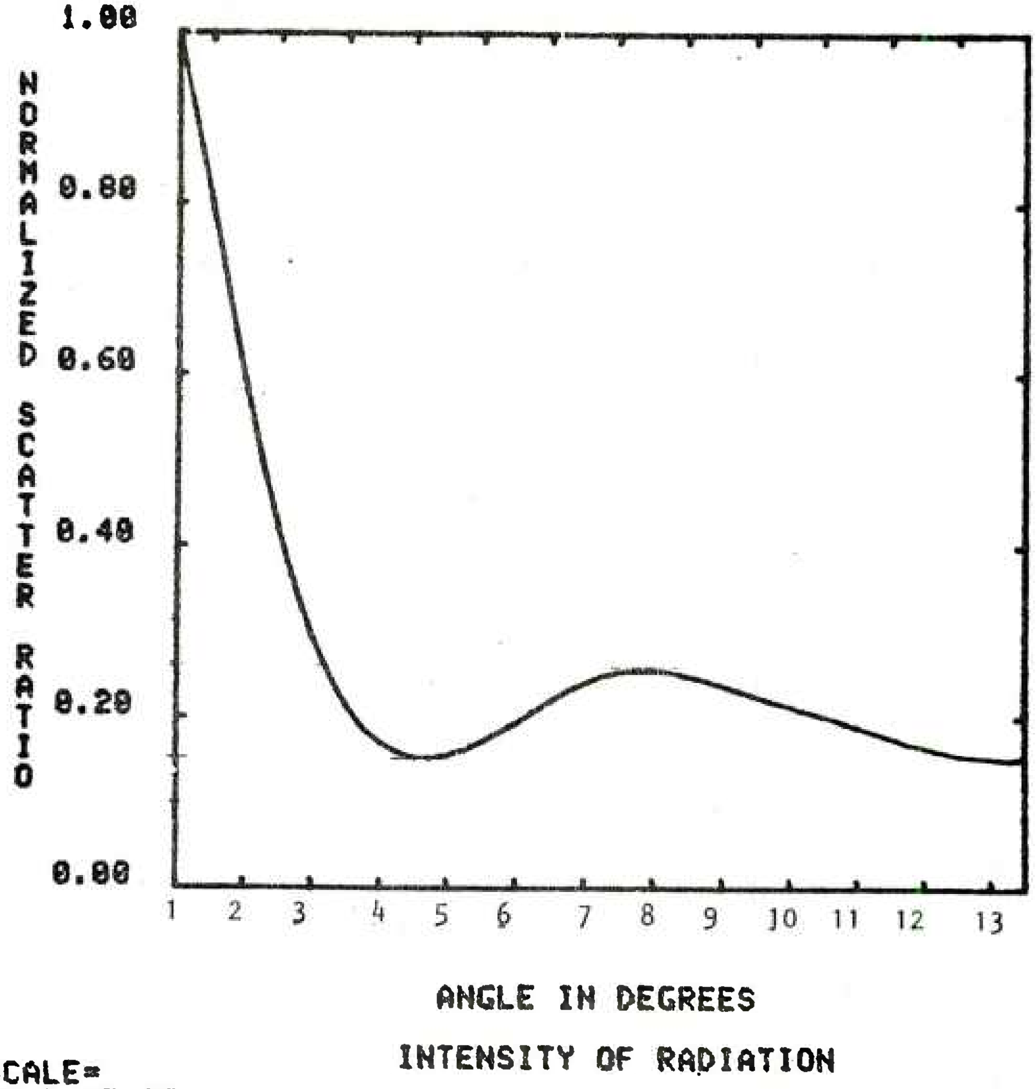
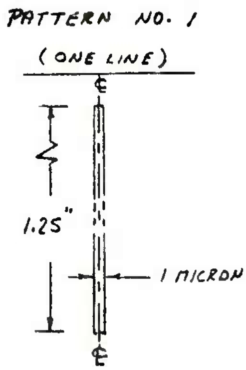


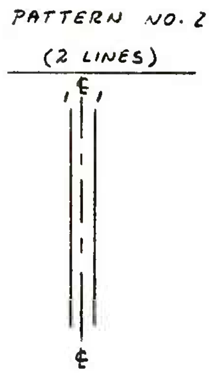
Figure 4. Power scattered at ±5 and ±10 as a function of scratch number. Open circles are averages of 5 artifacts (10 points); closed circles are extrema.



SCALE=
1.349E-004
ALPHA=5
BETA=0.45
INCOH PF=
3.998E-001
GROOVE COUNT=
10
Z2=0.1
C=1.065
INDEX=1.5
WAVELENGTH=
0.55
D0=2.75

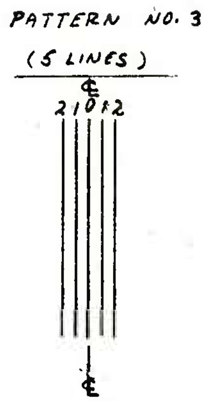
Figure 5. Theoretical scattering pattern from proposed grating configuration





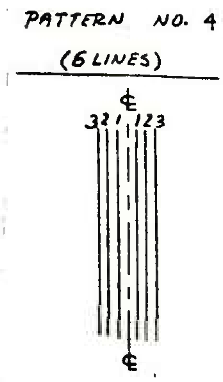
LINE SPACING

FROM PATTERN ℄ TO LINE NO.	DISTANCE (MICRONS)
1	2.14



LINE SPACING

FROM LINE NO. 0 TO LINE NO.	DISTANCE (MICRONS)
1	4.66
2	10.16

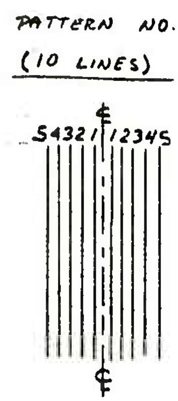


LINE SPACING

FROM PATTERN ℄ TO LINE NO.	DISTANCE (MICRONS)
1	3.03
2	9.96
3	18.05

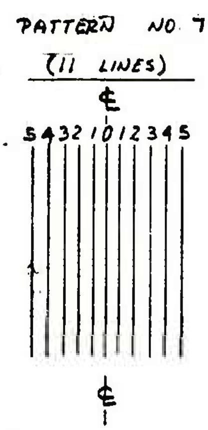
LINE SPACING

FROM LINE NO. 0 TO LINE NO.	DISTANCE (MICRONS)
1	7.50
2	16.37
3	26.60



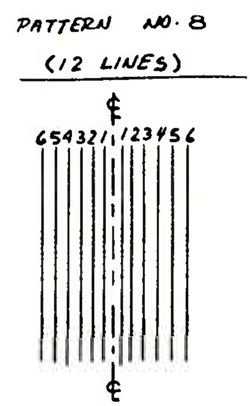
LINE SPACING

FROM PATTERN ℄ TO LINE NO.	DISTANCE (MICRONS)
1	1.44
2	4.74
3	8.59
4	13.00
5	17.94



LINE SPACING

FROM LINE NO. 0 TO LINE NO.	DISTANCE (MICRONS)
1	2.87
2	6.27
3	10.19
4	14.63
5	19.59



LINE SPACING

FROM PATTERN ℄ TO LINE NO.	DISTANCE (MICRONS)
1	1.30
2	4.27
3	7.73
4	11.69
5	16.15
6	21.10

Figure 6. Line spacing specifications

PATTERN NO. 9

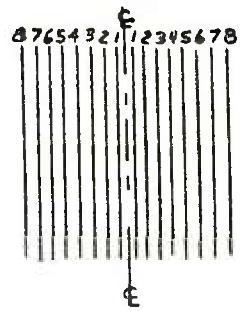
(13 LINES)

LINE SPACING

FROM LINE NO. 0 TO LINE NO.	DISTANCE (MICRONS)
1	2.57
2	5.61
3	9.11
4	13.09
5	17.53
6	22.44

PATTERN NO. 10

(16 LINES)

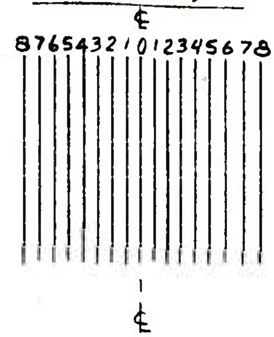


LINE SPACING

FROM PATTERN ℄ TO LINE NO.	DISTANCE (MICRONS)
1	1.07
2	3.47
3	6.19
4	9.24
5	12.62
6	16.34
7	20.38
8	24.75

PATTERN NO. 11

(17 LINES)

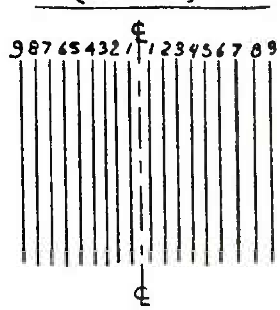


LINE SPACING

FROM LINE NO. 0 TO LINE NO.	DISTANCE (MICRONS)
1	2.21
2	4.73
3	7.55
4	10.67
5	14.09
6	17.82
7	21.84
8	26.18

PATTERN NO. 12

(18 LINES)

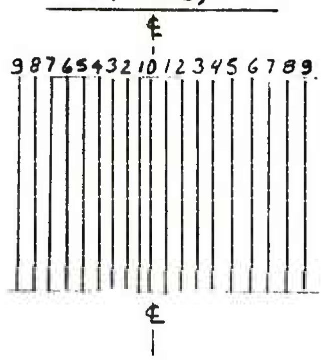


LINE SPACING

FROM PATTERN ℄ TO LINE NO.	DISTANCE (MICRONS)
1	1.00
2	3.20
3	5.67
4	8.42
5	11.45
6	14.75
7	18.32
8	22.17
9	26.30

PATTERN NO. 13

(19 LINES)

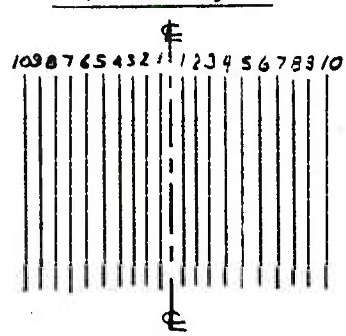


LINE SPACING

FROM LINE NO. 0 TO LINE NO.	DISTANCE (MICRONS)
1	2.34
2	4.95
3	7.84
4	11.00
5	14.44
6	18.15
7	22.14
8	26.40
9	30.94

PATTERN NO. 14

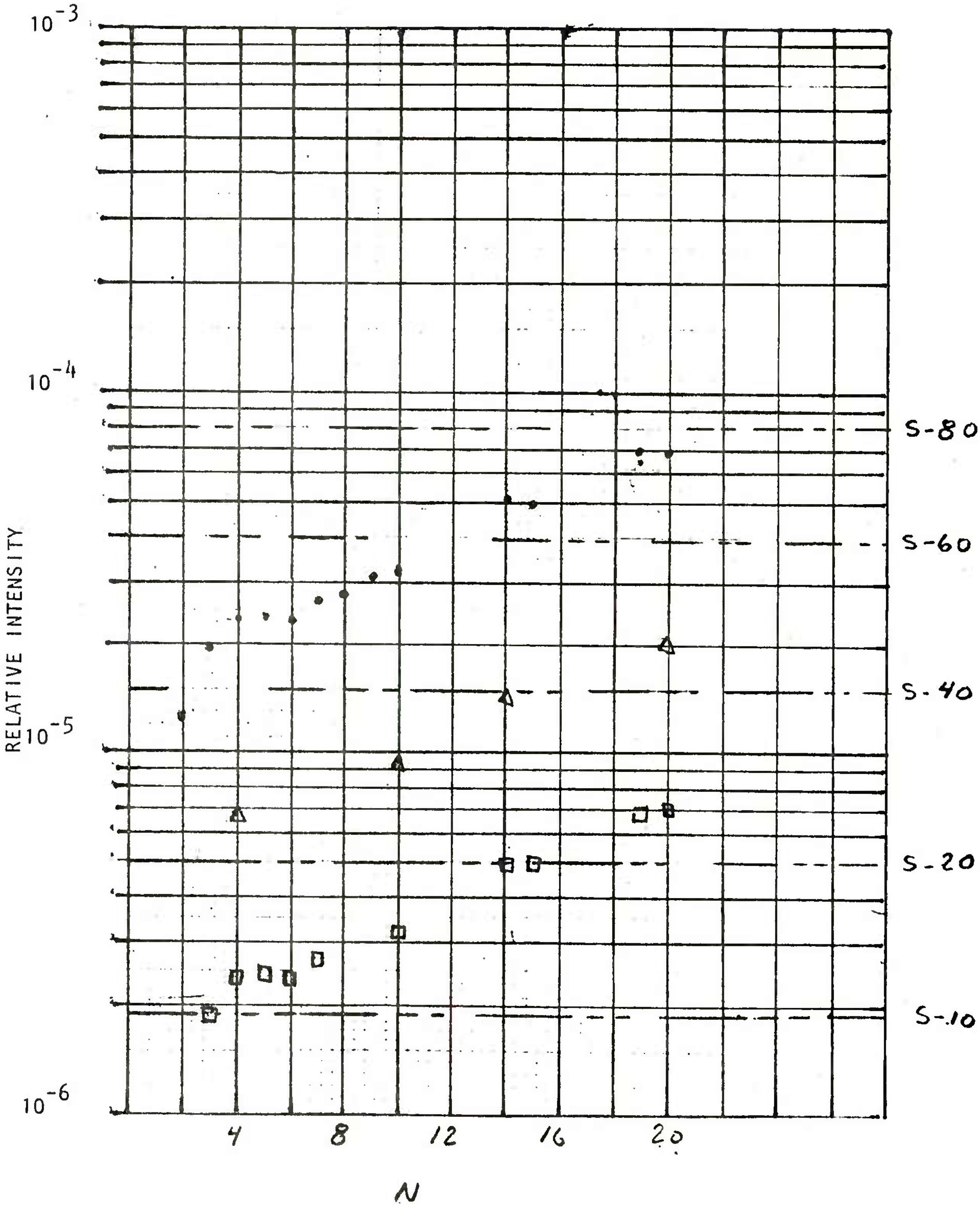
(20 LINES)



LINE SPACING

FROM PATTERN ℄ TO LINE NO.	DISTANCE (MICRONS)
1	1.16
2	3.68
3	6.46
4	9.51
5	12.82
6	16.40
7	20.23
8	24.34
9	28.70
10	33.34

Figure 6. (cont)



• Calculated for Z = 0.10 μm

Δ Calculated for Z = 0.05 μm

□ Calculated for Z = 0.03 μm

Figure 7. Peak-scattered intensity

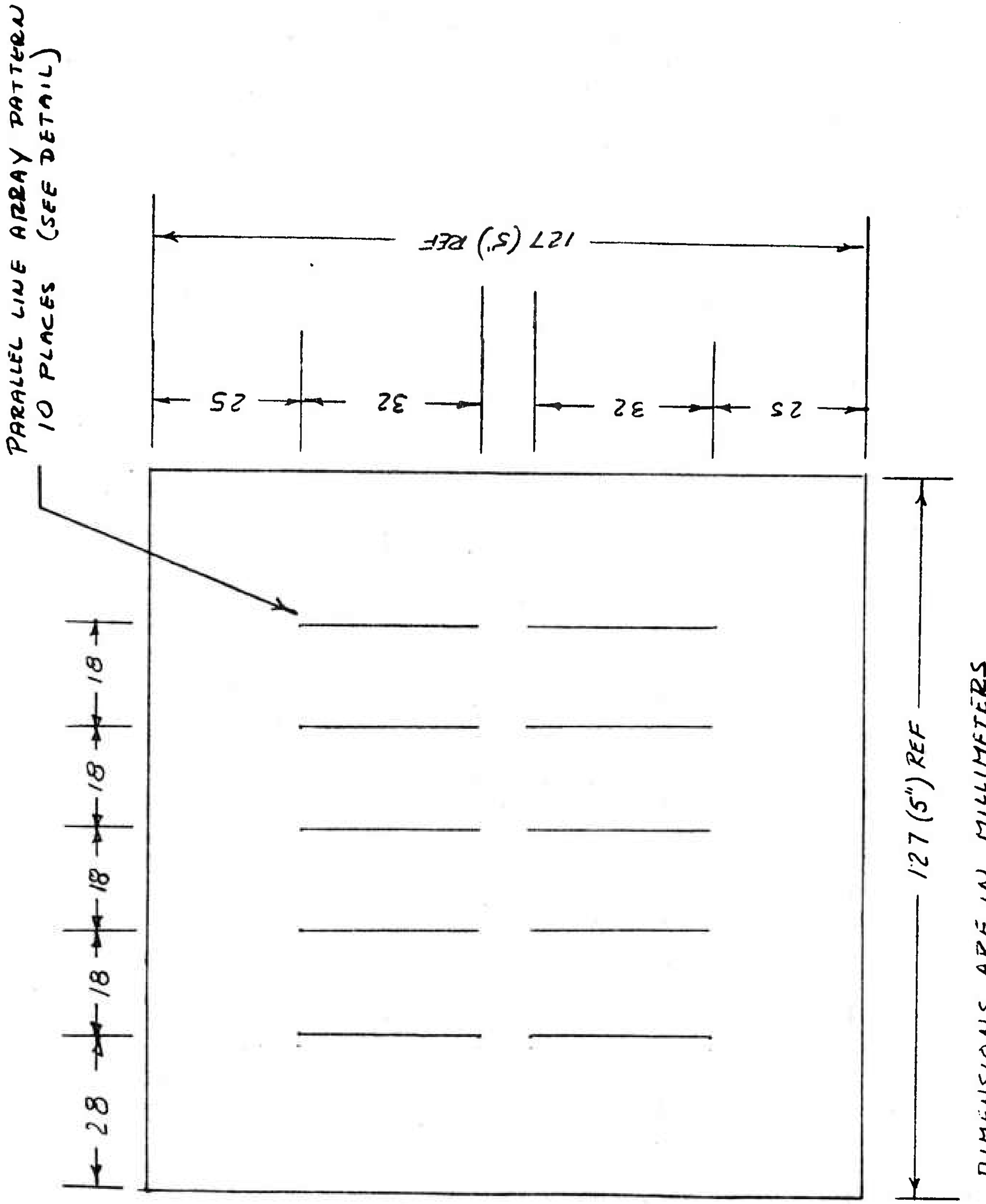


Figure 8. Configuration of scratch patterns for production processing

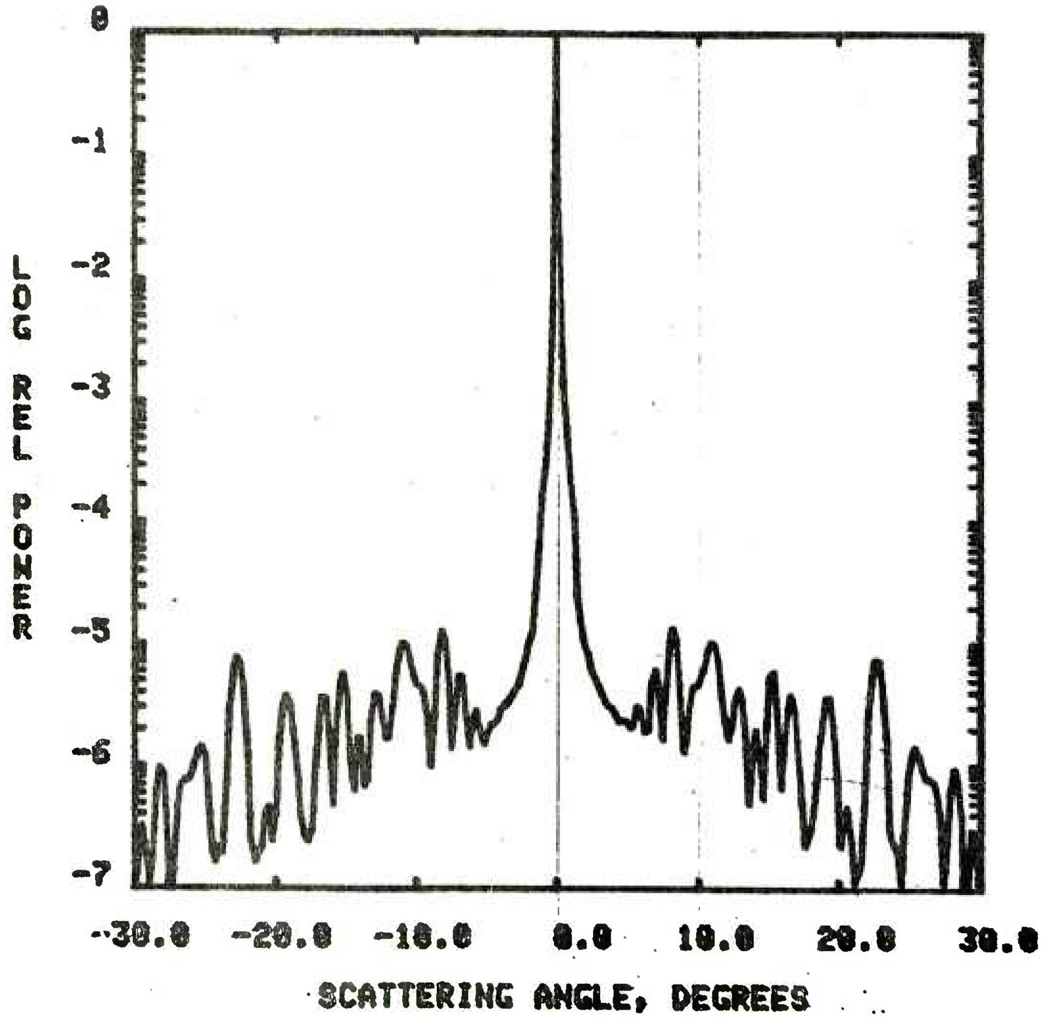


Figure 9. Measured scattering intensity for chemically etched S-20 standard

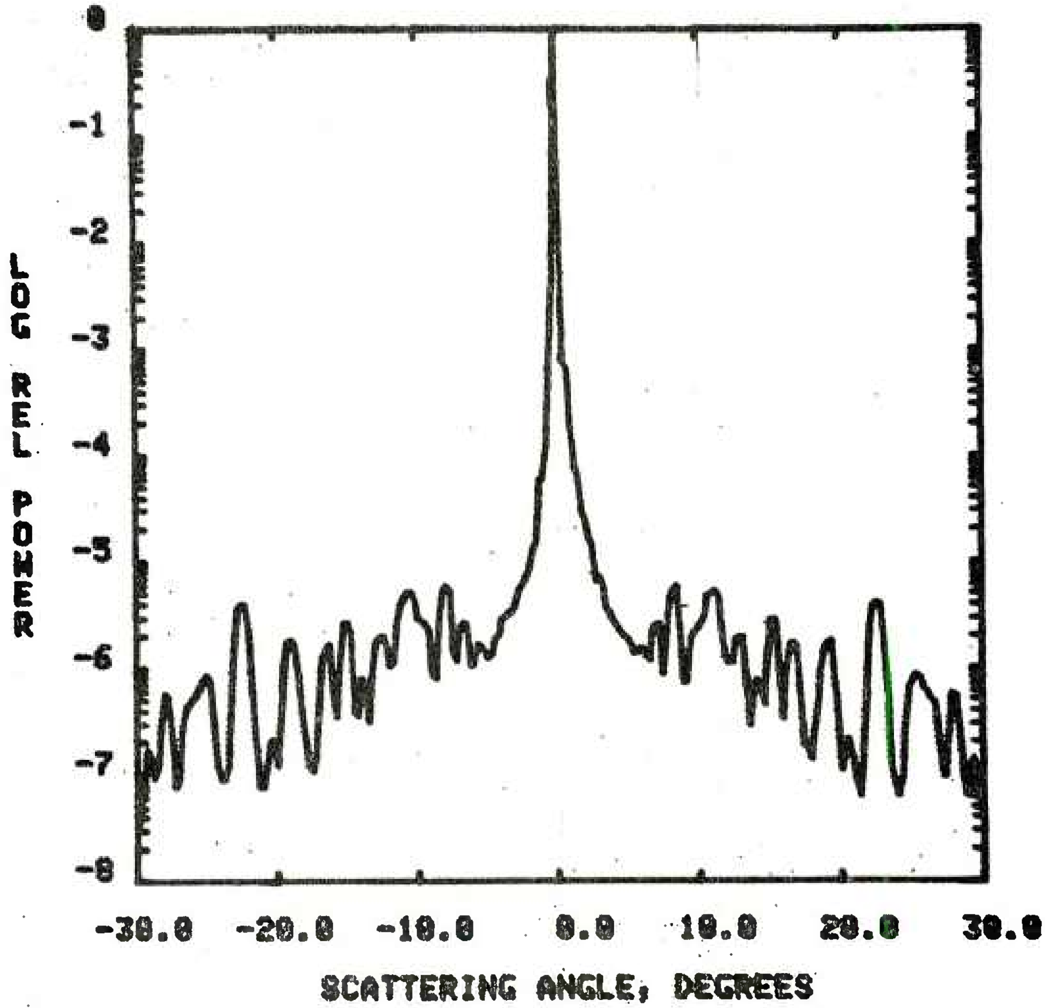


Figure 10. Measured scattering intensity for ion beam-etched S-20 standard

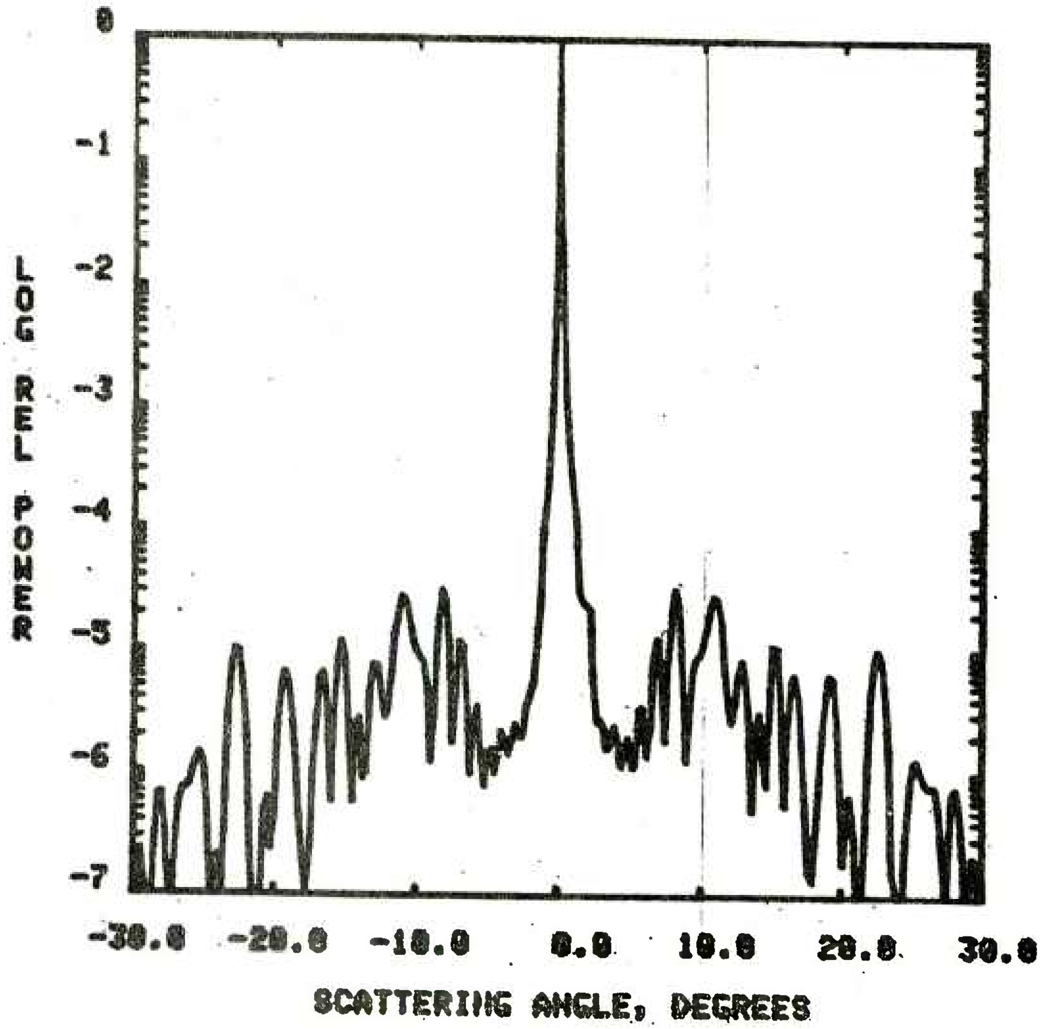


Figure 11. Measured scattering intensity for chemically etched S-40 standard

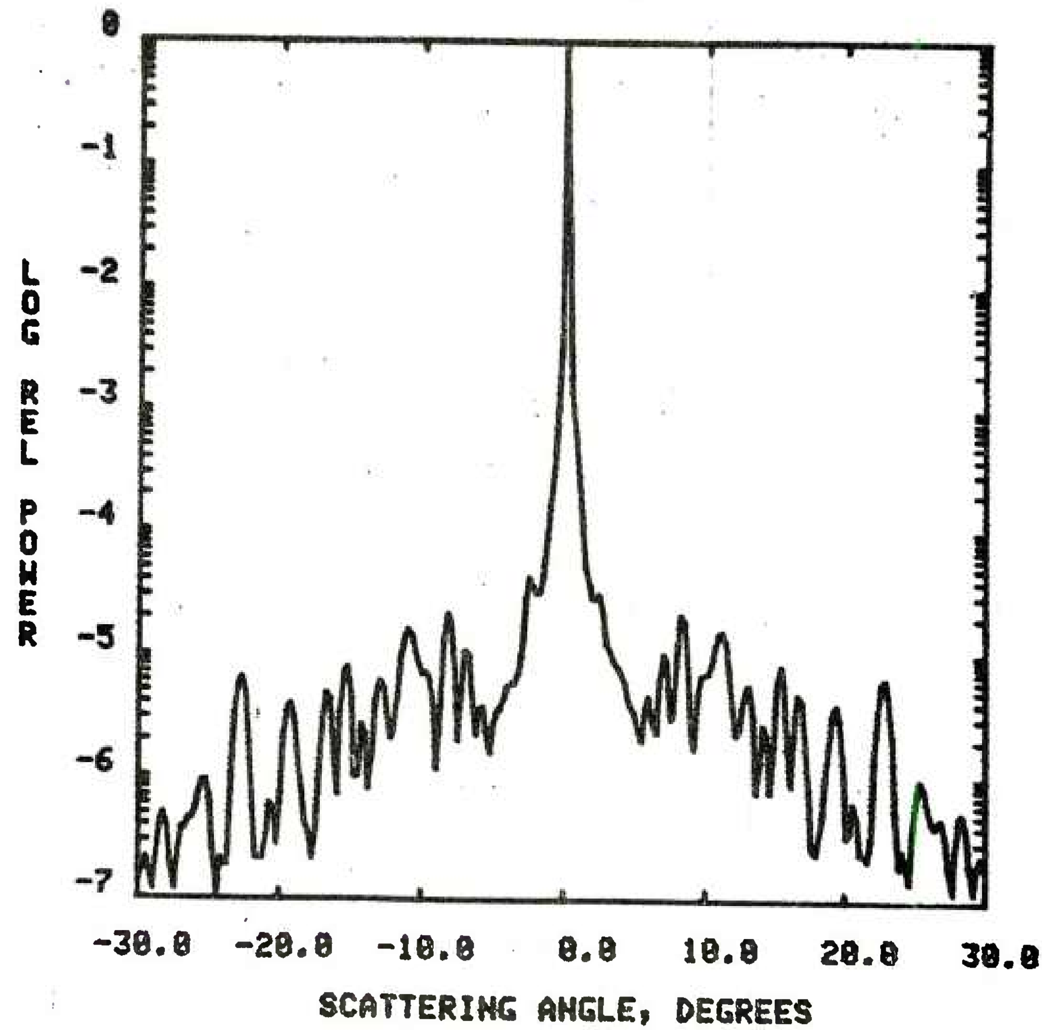


Figure 12. Measured scattering intensity for ion beam etched S-40 standard

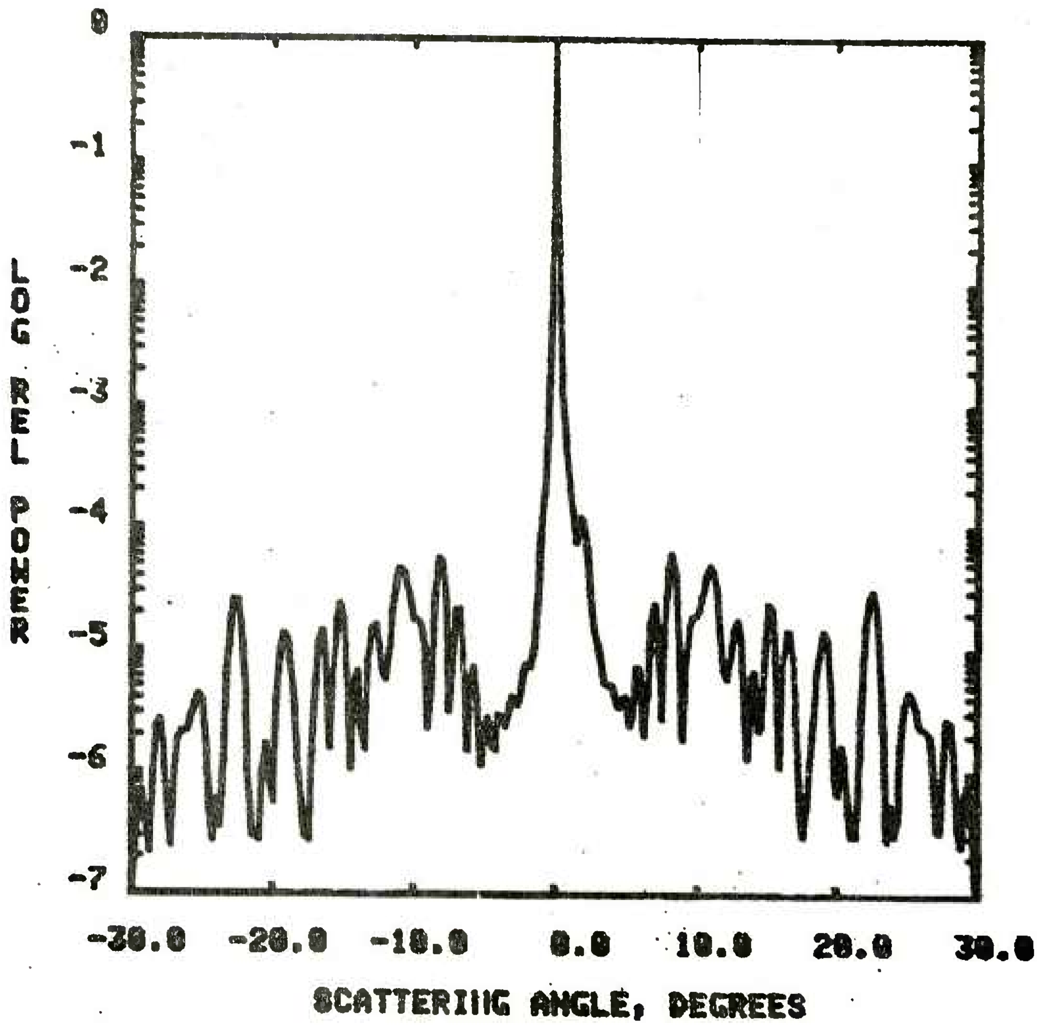


Figure 13. Measured scattering intensity for chemically etched S-60 standard

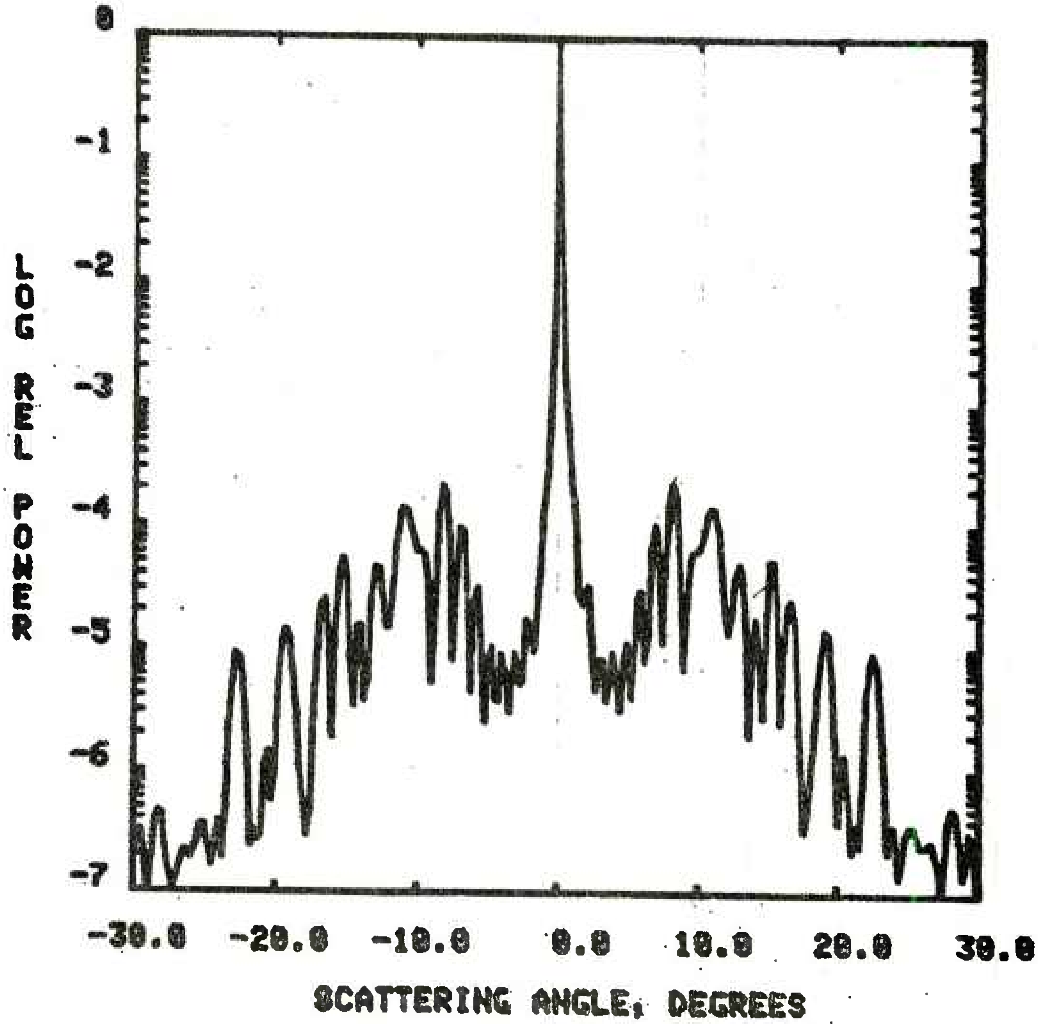


Figure 14. Measured scattering intensity for chemically etched S-80 standard

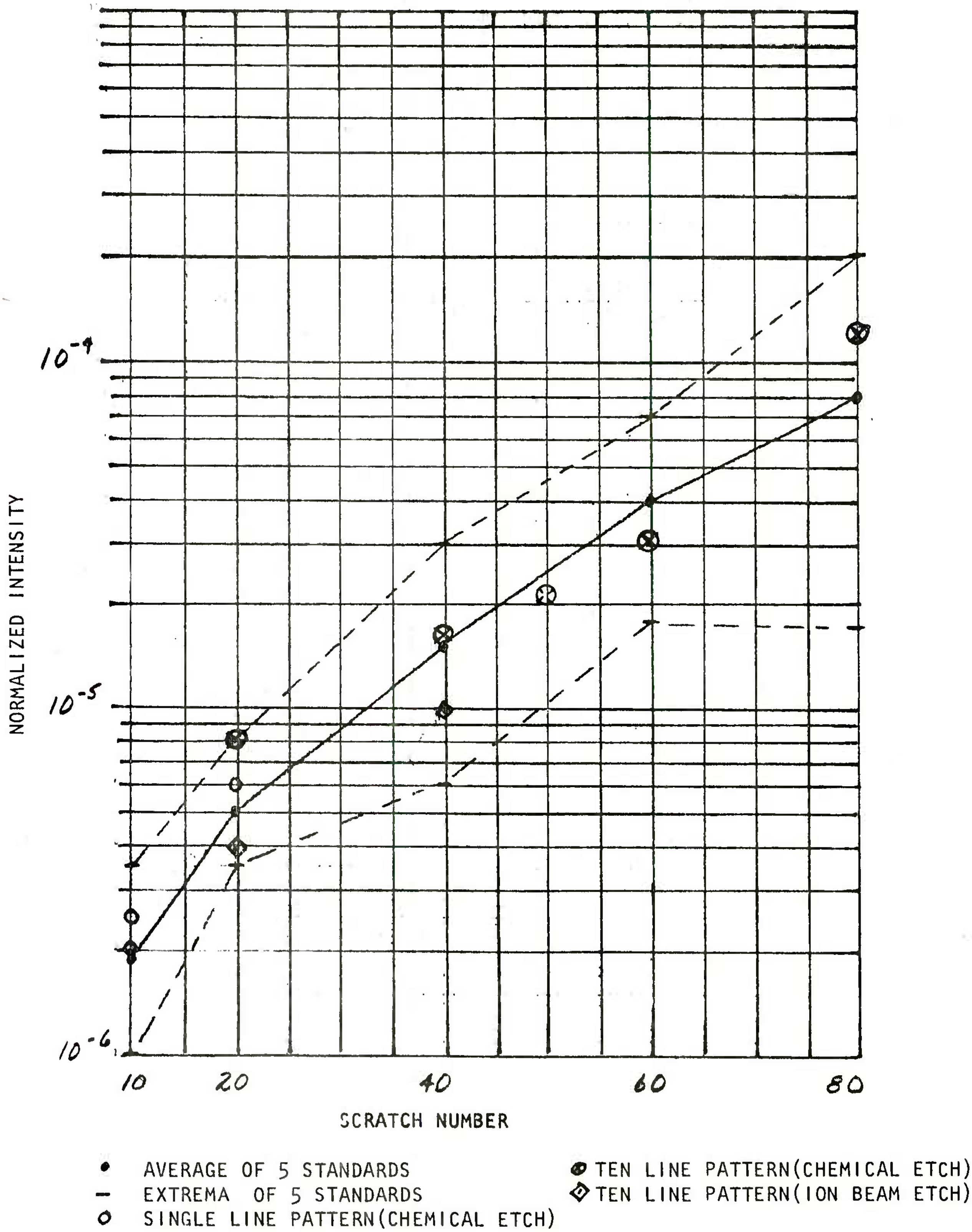


Figure 15. Correlation of scattered intensity and scratch number

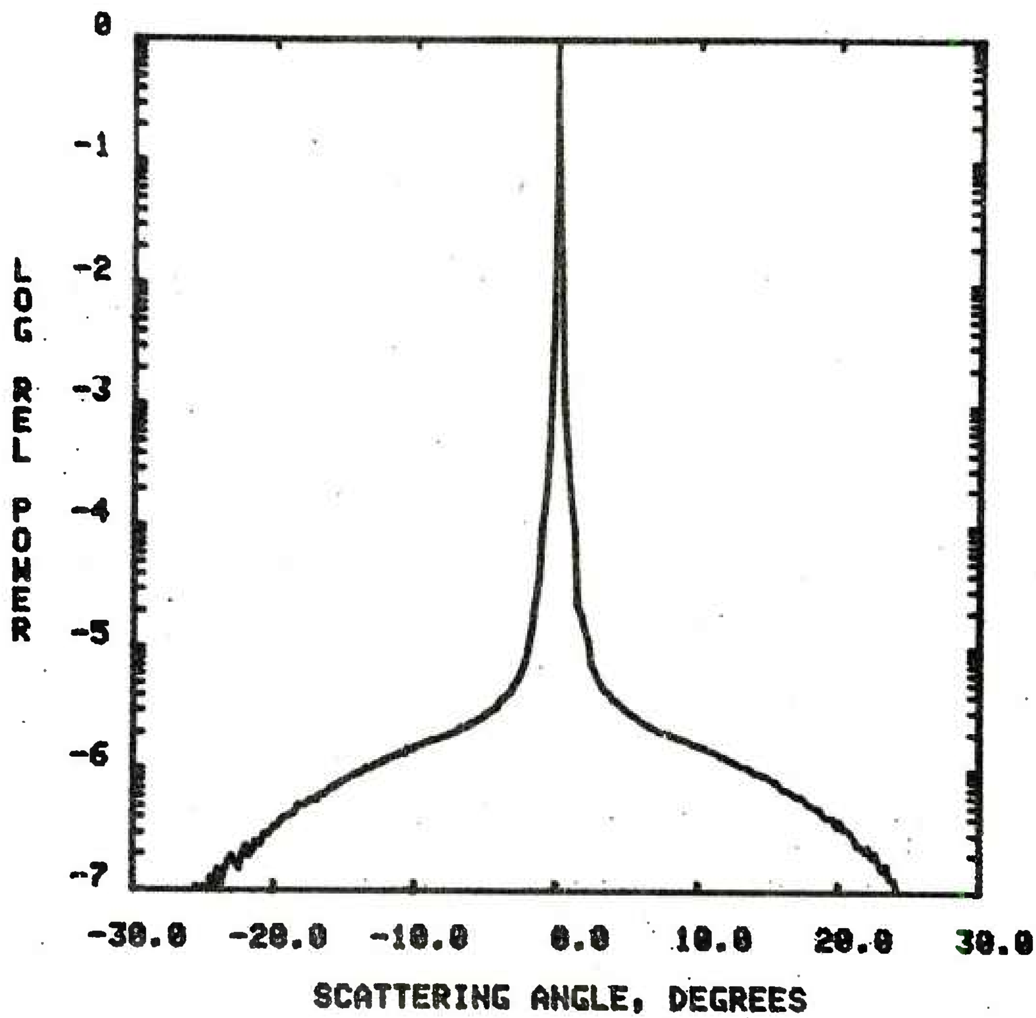
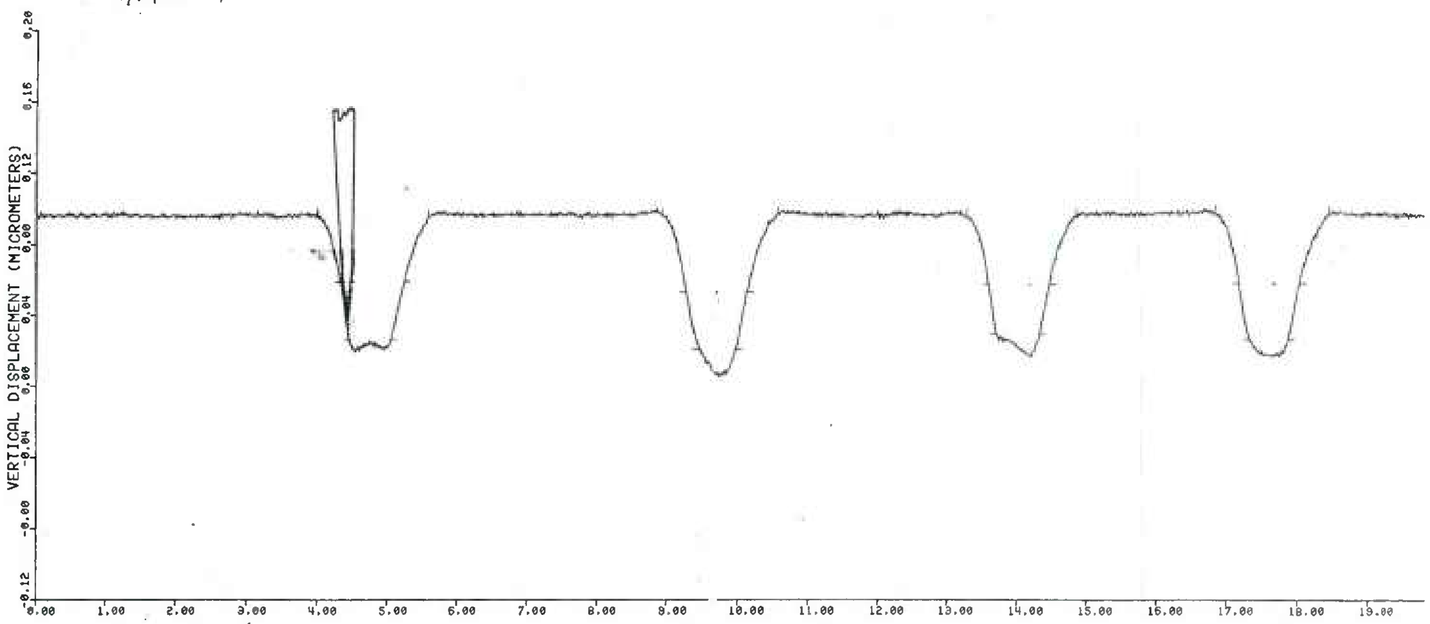


Figure 16. Measured scattered intensity of a chemically etched single-groove artifact

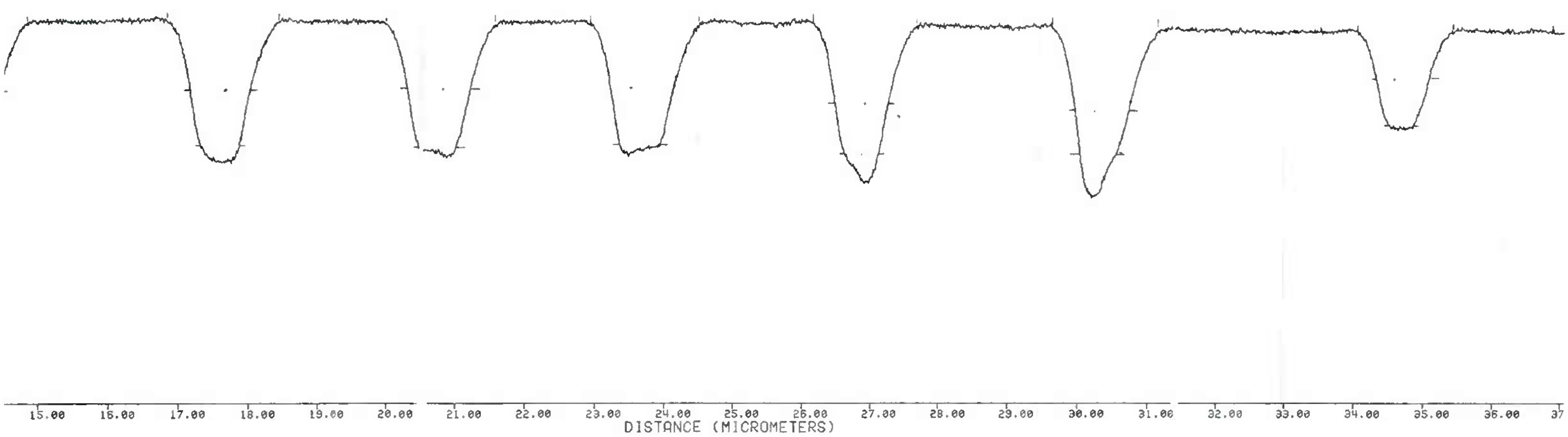
DECILOG GLASS SUBSTRATE 4Q2

PROF NO = 4   PTS 1   TO 4000

G14NOV84



(A)



15.00 16.00 17.00 18.00 19.00 20.00 21.00 22.00 23.00 24.00 25.00 26.00 27.00 28.00 29.00 30.00 31.00 32.00 33.00 34.00 35.00 36.00 37
DISTANCE (MICROMETERS)

Figure 1

B

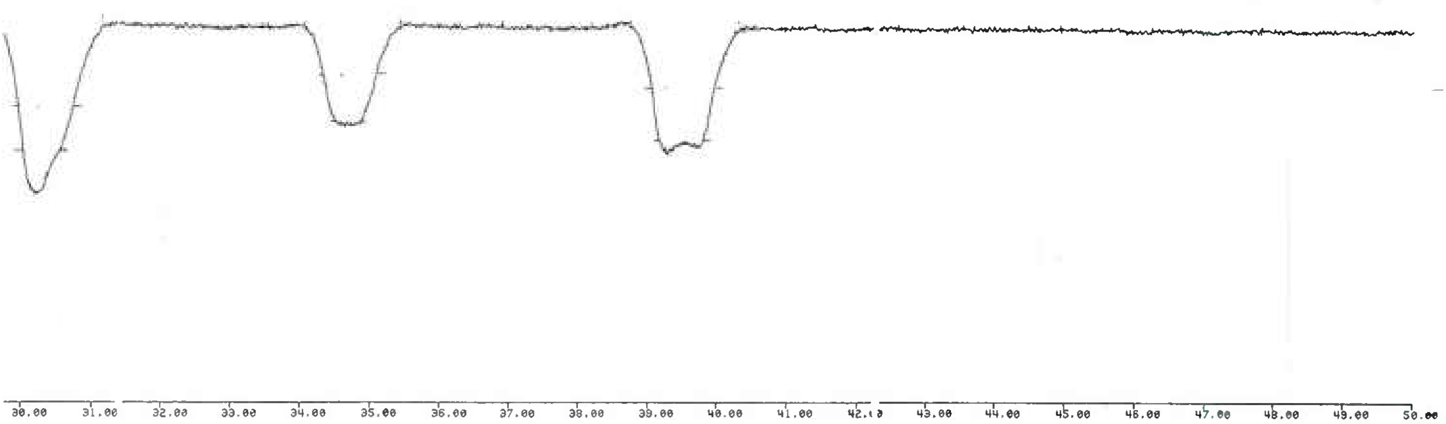


Figure 17. Profilometer scan of multi-channel artifact

C

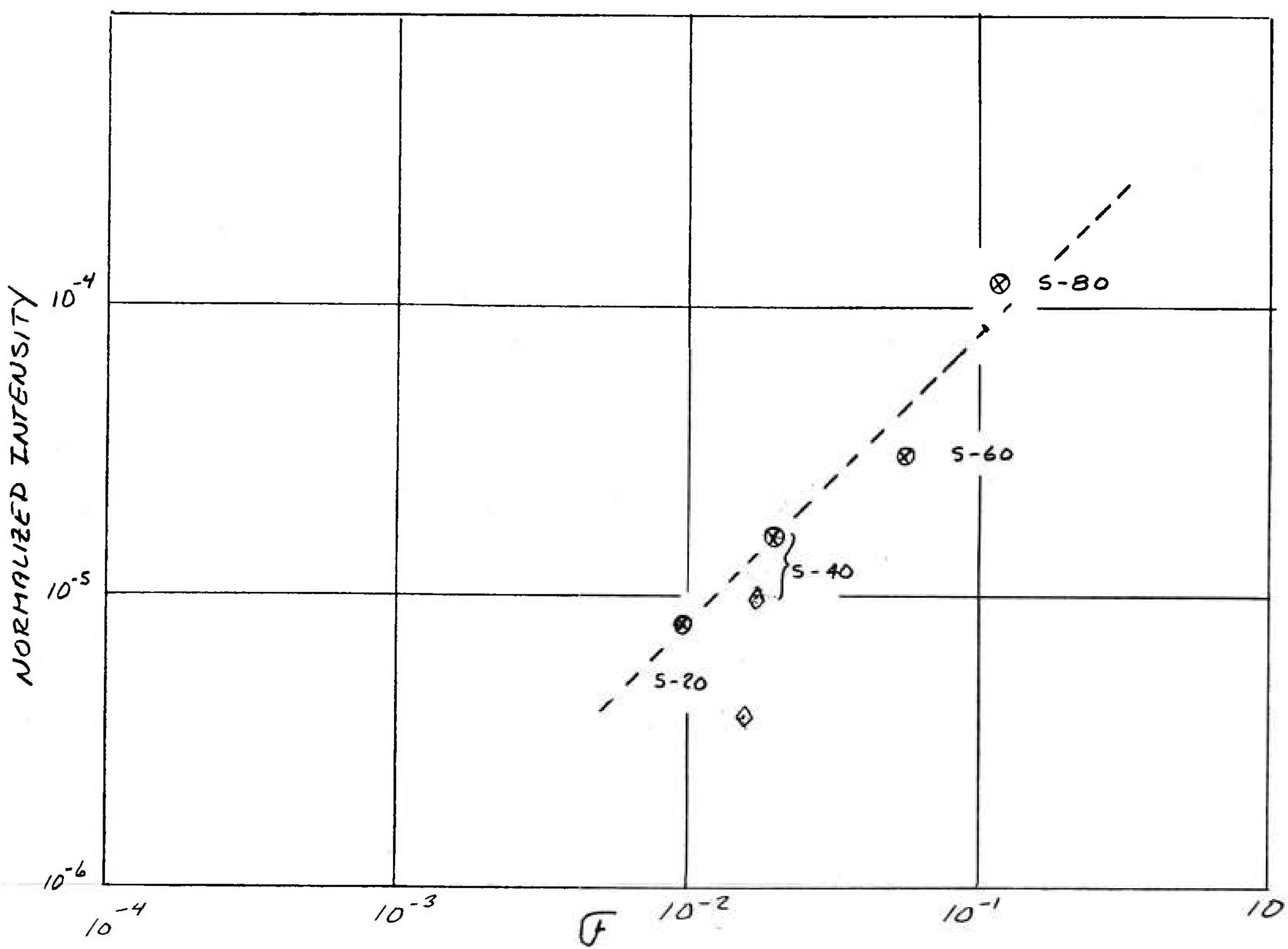


Figure 18. Correlation between scattered intensity and groove geometry

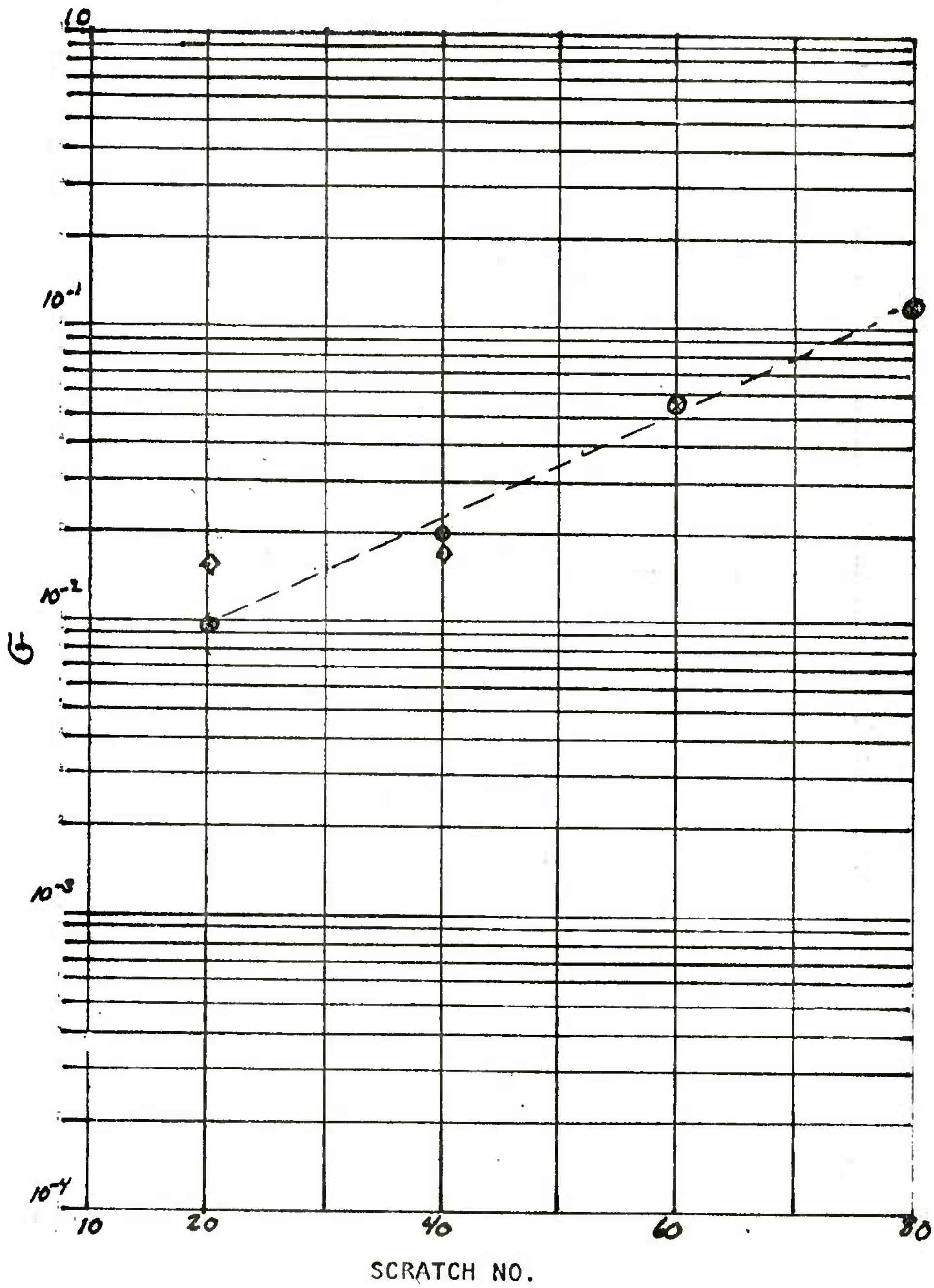


Figure 19. Correlation of groove geometry with scratch number

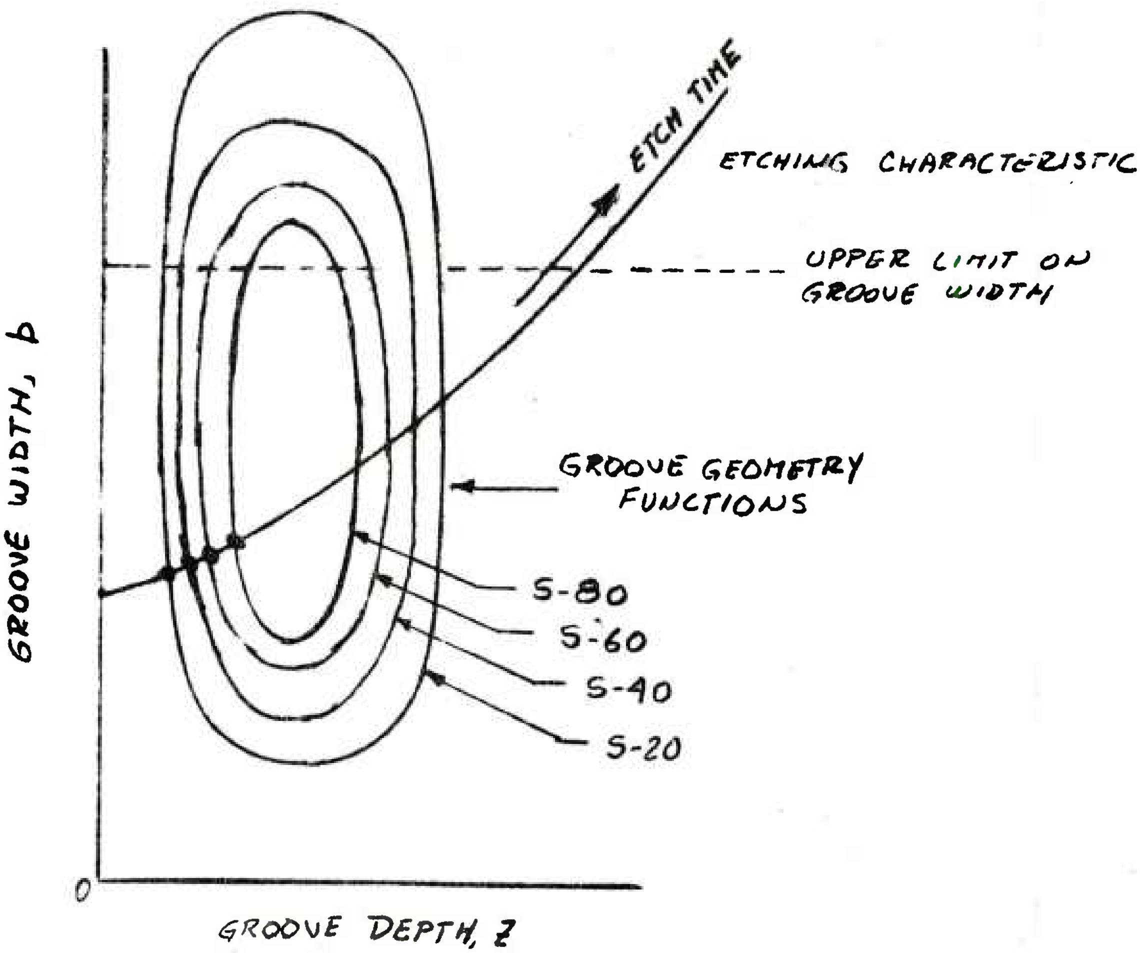


Figure 20. Method for determining etch times for scratch standards

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